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DEQ Review Draft

Remedial Investigation/Feasibility Study Work Plan for the Portland Shipyard

Prepared for Port of Portland

January 28, 2000

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IN ASSOCIATION WITH

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AND

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INTRODUCTION

Project Purpose and Scope

The purpose of this document is to satisfy a request made by the Oregon Department of Environmental Quality (DEQ) for the Port of Portland (Port) to conduct a remedial investigation (RI) and feasibility study (FS) at the Portland Shipyard (PSY). This is the next step in a process that the DEQ and Port started in late 1998 when DEQ issued its Strategy Recommendation for the PSY. About this same time, the Port provided DEQ with the Portland Shipyard Sediment Investigation Report (Striplin, 1998), a report documenting the results of a comprehensive sediment investigation for the PSY, and provided a briefing on the Port's preliminary conceptual site model for the PSY. This briefing was followed in March of 1999 by DEQ's Draft File Review Memorandum for the PSY. The Port provided comments on the Draft File Review Memorandum in May of 1999 and submitted a series of consultant reports documenting past upland site investigation and remediation activities. In December of 1999, the Port provided DEQ with additional information on the history of dredging and dredge material disposal at the PSY for purposes of supplementing and correcting DEQ's File Review Memorandum.

The scope of this work plan includes all of the principal steps in the RI/FS process: remedial investigation, risk assessment (both human health and ecological), and feasibility study. The scope of this work plan will also include the identification of sources of contamination to the PSY and parties who contributed to the contamination of the PSY, including current and past operators, contractors and tenants; Kaiser Company, Inc.; the Federal government; potential upstream and downstream sources; and potential sources within the Swan Island Lagoon such as the City of Portland outfalls, Coast Guard facility, U.S. Navy facility and others.

Site Location and Description

The PSY address is 5555 North Central Channel Avenue in Multnomah County, Portland, Oregon. The PSY is located in north Portland, between Swan Island Lagoon and the Willamette River on the peninsula known as Swan Island (Figure 1).

Figure 2 shows the boundaries of the "Site" for purposes of the RI/FS. The Site includes 94 acres of uplands and 106 acres of submerged lands owned by the Port. The Site also includes Berth 311, a concrete pier/lay berth located on the east side of Swan Island Lagoon, also owned by the Port. Adjacent to the Site are submerged lands owned by the Oregon Division of State Lands (ODSL). These lands include the main channel of the Willamette River and Swan Island Lagoon. The Port currently leases

two parcels of submerged lands in Swan Island Lagoon from ODSL: approximately 14 acres between Berth 301 and Berth 308 and approximately 4.5 acres at Berth 311 (see Figure 2).

Current operations at the PSY consist of the repair and maintenance of privately owned and government vessels from the United States and overseas. Ship repair and maintenance are conducted in three dry docks (i.e., Dry Docks 1, 3 and 4) and 15 berths along the perimeter areas of the shipyard. The dry docks are used for raising vessels out of the water to perform hull repair, maintenance, painting, and other dry lay-up ship repair tasks.

The berths are outfitted with 16 electrically powered cranes in a variety of sizes and lift capacities. The cranes operate on tracks that are laid along the berths and allow them mobility between the berths. The berths are also used for maintenance that does not require lifting the vessel out of the water. This includes cleaning tanker vessel ballasts, engine maintenance, outfitting, deck painting, and other activities.

The shipyard's upland areas, or yard, house the support services for both ship repair operations and maintenance of the shipyard infrastructure. The current activities or operations in the shipyard's upland areas include:

- Metal machining (for facilities maintenance and ship repair)
- Carpentry
- Electrical shop
- Steel fabrication
- Propeller repair and services
- Mobile equipment maintenance
- Paint storage and painting operations
- Abrasive blasting (grit and steel shot) and surface preparations
- Berth and ship rigging storage and support

The ballast water treatment plant (BWTP) accepts oily waste from vessel cleaning and maintenance. The plant receives oil water slops from tankers and other vessels. The slops are temporarily stored in the tanks, and the oil is extracted by natural separation, addition of chemicals, and heating of the wastewater. The reclaimed oil is recycled. The treated water is either pumped to the city sewer system or discharged to the Willamette River, depending upon analytical results. The BWTP is a critical marine facility within the Portland Harbor in that the Coast Guard requires that facilities be available to manage oily water and ballast water for ships entering waters of the United States.

The PSY has a storm water treatment system to treat water from the dry docks. The storm water treatment system uses chemical and mechanical processes to remove contaminants.

Work Plan Documents

This work plan consists of a set of documents that will guide the RI/FS. It has been developed in general accordance with OAR 340-122, DEQ guidance, and EPA guidance (EPA, 1988). The individual elements of the work plan are briefly summarized below;

Work Plan – The main text of the Work Plan describes the physical setting, site history and conceptual site model. It also presents the site characterization, risk assessment, and feasibility study plans. The Work Plan includes supporting tables and figures.

Project Management Plan (PMP) – The PMP (Appendix A) includes a description of the project team members and their roles; a list of submittals; a project schedule; and procedures for addressing deviations from the Work Plan.

Sampling and Analysis Plans (SAP) – A SAP is a detailed description of the scope of the RI and procedures that will be used to collect samples and complete chemical analyses. It includes quality assurance/quality control (QA/QC) procedures for both the field and laboratory. The SAP is intended to serve as a manual for field staff. Appendix B contains the SAP for soil and groundwater sampling activities. Appendix C contains that SAP for surface water and sediment sampling activities.

Health and Safety Plans (HSP) – A HSP presents a description of procedures to be used in the field to protect personnel from potential hazards that may exist during on-site activities. Appendix D contains the HSP for the soil and groundwater sampling subcontractor, Hahn and Associates. Appendix E contains the HSP for the surface water and sediments sampling subcontractor, Striplin Environmental Associates.

PHYSICAL SETTING

Site Location and Description

The PSY is located near the western boundary of the Portland Basin. The Basin is bounded on the west and southwest by the Tualatin Mountains and on the east and southeast by the foothills of the Cascades. The Site is bounded by Swan Island Lagoon to the north and east, the Willamette River to the north and west, and a variety of industrial operations to the south.

Most of the Site is covered with buildings or pavement. The only substantive unpaved area is the N. Channel Avenue Fabrication Site located on the southeast portion of the site between N. Channel Avenue and the Willamette River. Along Swan Island Lagoon, the berths are supported by pilings that extend into the bank. The bank underlying the berths slopes into the lagoon. On the Willamette River side of the site and near the dry docks, the river shore slopes toward the river or is contained behind sheet piling.

Topography

Based on the United States Geological Survey (USGS) Portland Quadrangle Oregon-Washington 7.5-Minute Series Topographic Quadrangle Map, the Site is located approximately 40 feet above mean sea level, and the site is generally flat.

Bathymetry

Figure 3 illustrates the bathymetry of the Willamette River near the PSY based on measurements made by the U.S. Army Corps of Engineers (COE) in 1998 as part of their proposed channel deepening project. The river bottom slopes toward the navigation channel which ranges in depth from approximately –60 feet (NGVD, 1947), near Dry Dock 4, to –39 (NGVD, 1947) feet in the navigation channel across from Berth 315.

Based on a hydrographic survey conducted by the Port in 1989, river bottom elevations in the Swan Island Lagoon range from approximately -24 feet (Columbia River Datum, CRD) adjacent to the berths to -38 feet (CRD) near the center of the Lagoon across from the Berth 301.

The design elevations for the bottom of the three dry dock basins are -48 feet CRD, -57 feet CRD, and -65 feet CRD for Dry Docks 1, 3 and 4, respectively. Hydrographic surveys conducted by Cascade General in May of 1996 indicate that the approximate current bottom elevations of the Dry Docks 1, 3 and 4 are -50 feet CRD, -59 feet CRD, and -60 feet CRD, respectively.

Climate

The Portland area has a temperate marine climate characterized by wet winters and dry summers. Precipitation, temperature, and wind data for the site are summarized below.

Precipitation (Hart Crowser, 1998)

Average Annual	37 inches (mostly rain)
Wettest Months	November through February (greater than 4 inches per month)
Driest Months	July and August (less than 1 inch per month)

Temperature (Hart Crowser, 1998)

Average Annual	54 °F
Coldest Month	January (Average 40 °F)
Warmest Month	August (Average 69 F)
Lowest Recorded	-2 F (January 1888)
Highest Recorded	107 F (July 1942)

Wind (Hart Crowser, 1998)

Minimum Monthly Mean (October): 6 mph

Maximum Monthly Mean (December/January): 10 mph

Maximum Recorded: 88 mph

Direction: Generally from west, but east winds are common

Regional Geology and Hydrogeology

The following discussion was taken from Swanson et al. (1993) and Hartford and McFarland (1989).

Geology

The Site is located in the Portland Basin, a northwest-southeast trending basin that is approximately 20 miles wide by 45 miles long. This structural basin is filled with consolidated and unconsolidated continental sedimentary rocks. Older rocks that underlie the basin-fill sediments include the Skamania Volcanics; Columbia River Basalt Group; basalts of the Waverly Heights, Goble Volcanics, Pittsburg Bluff, Scappoose, and the Rhododendron Formations. The Sandy River Mudstone and the Troutdale Formation are the oldest of the basin filling-sediments. Large quantities of Pleistocene sediments were deposited during catastrophic floods of the Columbia River. These floods occurred as a result of the periodic failures of ice dams impounding huge lakes in Idaho and Montana. The catastrophic flood

deposits can be grouped into two easily discernable lithologic units: a basaltic sand and gravel unit with varied amounts of cobbles and boulders and a finer, stratified, micaceous arkosic sand, silt and clay. The former unit is present near the Columbia River channel in southern Clark County and north Portland. The later unit exists at an altitude of about 250 feet throughout the Portland Basin. Alluvium deposits from the Columbia were deposited on the Pleistocene sediments. The alluvium deposits consist of sand and silt.

Hydrogeology

Eight major hydrogeologic units form the Portland Basin aquifer system. Proceeding from oldest to youngest, these units include:

- Older rocks
- Sand and Gravel Aquifer (SGA)
- Confining Unit 2 (CU2)
- Troutdale Sandstone Aquifer (TSA)
- Confining Unit 1 (CU1)
- Unconsolidated sedimentary rock aquifer
- Consolidated gravel aquifer
- Undifferentiated fine-grained sediments

Of these, the consolidated gravel aquifer and the undifferentiated fine-grained sediments are the two hydrogeologic units that are relevant to the Site.

Consolidated Gravel Aquifer

The consolidated gravel aquifer is composed of several geologic formations that consist of cemented conglomerate and sandy conglomerate, as well as local accumulations of lavas and surface soils. The conglomerate portion of the aquifer, referred to as the Troutdale Gravel Aquifer (TGA), extends throughout the Basin. The top of the consolidated gravel aquifer typically ranges from plus 100 to 200 feet. Near the Willamette and Columbia Rivers, the top of the aquifer is eroded to a depth of approximately minus 200 feet. The TGA is an source of public, industrial, and domestic supply. Most wells in the TGA yield a minimum of 50 gallons per minute (gpm) and can yield up to 1,000 gpm.

Unconsolidated Sedimentary Aquifer

The unconsolidated sedimentary aquifer is the uppermost hydrogeologic unit in the Basin. It consists of catastrophic flood deposits, alluvial deposits along smaller streams in the Basin, flood plain deposits along the Willamette and Columbia Rivers, and glacial outwash in basins in northern Clark County. Aquifer yields in the unconsolidated sedimentary aquifer are variable.

SITE HISTORY

PSY Development and Use

Prior to 1926

Swan Island was originally a periodically flooded sand bar and marsh with the main channel of the Willamette River between the island and Mocks Bottom to the east. The Willamette River on the west side of the island was too shallow for ship navigation. The main ship channel was east of the island. The Port purchased Swan Island in 1922. In 1923, the Port began a project to relocate the main channel of the Willamette River from the east side of the island to the west side of the island, and to raise the island to 32 feet above mean low water. In 1927, a causeway was built in the east channel from the mainland to the island, and the south end of Mocks Bottom was raised, making a peninsula of the island and creating a still water lagoon of the east channel. This lagoon is now referred to as Swan Island Lagoon.

Portland Municipal Airport (1926 to 1941)

In 1926, the Port began construction of a municipal airport at the site. Airport development was completed in 1931. The Site served at a municipal airport until operations ended in 1941. In 1942, airport operations were moved to the current location of Portland International Airport. The northwestern end of the landing field (runways and taxiways), administration building, and hangars were located on the portion of the former airport encompassed by the Site.

Kaiser Shipbuilding Facility (1942 to 1947)

In 1942, the Port leased the Site to the U.S. Maritime Commission for a period of 3 years. The Maritime Commission then contracted with Kaiser Company, Inc. for the acquisition, construction, and operation of shipyard facilities. In the same year, Kaiser started shipyard construction including dredging a "basin" in the Willamette River for purposes of operating a dry dock (currently Dry Dock 1). See Figure 2 for the location of Dry Dock 1 and other Site features. During the war emergency, Type T2 tanker (Liberty Ships) were built.

In 1945, the lease between the Port and the Maritime Commission was extended until 1952. Kaiser used the Site for ship repair and other industrial enterprises until 1947.

Operations by War Assets Administration (1947 to 1949)

In 1947, the War Assets Administration, the successor to the Maritime Commission, declared the assets at the Site to be surplus. The War Assets Administration then leased building and property space to a number of tenants for industrial uses, such as steel fabrication and storage; ship dismantling; wood products manufacturing; equipment manufacturing; maritime supply sales; printing; chemical and soap storage; war surplus storage; fire extinguisher service and storage; paint storage; aluminum oil tank manufacturing; service station; sheet metal shop; roofing supply storage; and general office storage.

Ship Repair Facility (1949 to Present)

The War Assets Administration entered into negotiations with the Port that culminated in the sale of all improvements at the Site in 1949. In 1950, the Port began operations of the Swan Island Ship Repair Yard (currently the PSY). This included the dredging of another basin and installation of Dry Dock 2. In 1957, the Port dredged a third basin and installed Dry Dock 3. By 1964, the former airport terminal was torn down. Construction of shipyard facility additions and new structures continued during the late 1960s and early 1970s. Facility construction included a treatment plant to manage ballast water (now referred to as the Ballast Water Treatment Plant, BWTP). This plant was constructed in 1973 and replaced by the current plant in 1979.

Further site development and construction occurred in the mid-1970s. A new barge construction transfer system was built in 1975 near Dry Dock 3. A large employee parking lot was constructed in the south-central portion of the Site in 1977. The next year, a basin for Dry Dock 4 was dredged. Dry Dock 4 was placed into operation in 1979. During this same timeframe, the new side of the yard (referred to as the "new yard") and Berths 312 through 314 were constructed.

During the 1980s, major module fabrication by ARCO (Atlantic Richfield Company) was undertaken at the PSY to support oil exploration activities in Alaska. This activity continued until 1989. During this period, the only other substantive changes to the PSY included taking Dry Dock 2 out of service.

Between 1949 and 1996, the Port both owned and operated the PSY. A number of contractors performed ship repair activities and a number of tenants performed industrial operations in leased facilities.

Operations Transfer to Cascade General (1996)

In 1995, Cascade General was awarded the operations contract for the PSY. Starting on January 1, 1996, Cascade General took over all

operations, maintenance, tenant management, and environmental responsibility, including permit and reporting responsibilities, for the PSY.

Previous Investigations

Previous Upland Investigations

A series of upland site investigations have been conducted at the PSY. These investigations are summarized in the following reports:

- 1989 UST decommissioning near Berth 311
- 1990 investigation of the ARCO (Channel Avenue Fabrication) site
- 1991 subsurface investigation between Buildings 58 and 64
- 1992 environmental assessment soils investigation near the former Norvac storage areas
- 1992 polychlorinated biphenyl inventory for Building 60, electrical substation
- 1992 investigation of soils north of Building 50
- 1993 subsurface soil sampling
- 1998 risk-based closure of a heating oil UST at the Central Utility Building

In addition, in 1998 the Port and Cascade General jointly performed upland sampling in seven areas as part of the proposed sale of the PSY. Table 1 summarizes the upland site investigation conducted in 1998. At each sampling location, soils were sampled near the surface, typically the upper two feet, and just above the water table, typically at a depth of 16 to 18 feet below ground surface (bgs).

In the Main Parking Lot and Building 10 and Grit Silo Area, relatively few constituents were detected and the ones that were detected were at relatively low concentrations. The sampling results for these two areas are discussed below. Constituents were detected at higher concentrations in the other five areas. As a result, DEQ determined that these areas had data gaps that need to be filled during the RI. The sampling results for these five areas are discussed in the next section, Site Characterization Plan.

Main Parking Lot

The main parking lot is approximately 8 acres and is located on North Channel Avenue adjacent to the guard gate at the main entrance. Currently the main parking lot is paved with approximately 3 inches of asphalt.

Kaiser Company site layout drawings show that during World War II boiler fabrication occurred in the main parking lot area.

Two samples were collected in the main parking lot area and analyzed for total petroleum hydrocarbons (TPH), polychlorinated biphenyls (PCBs), and metals. Boring No. 1 was located approximately 220 feet southeast of the Time Office/Nurse Station building. Boring No. 7 was located 250 feet west-northwest of Building 81 and 175 feet from the fence along Channel Avenue. Groundwater was encountered at 17.5 feet bgs.

Table 2 provides the boring number, sample interval, soil description, and analytical parameters for samples collected in the main parking lot.

Table 3 provides the analytical data for the parameters that tested above the detection limits.

Building 10 and Grit Silo Area

This area is paved and located immediately south of Berth 304. Several roll-off boxes and a grit hopper with slag grit were visible on the ground in this area. This area has been historically used for grit storage and blasting and equipment storage, and most recently tank mucking.

The sample location, Boring No. 16, was 35 feet west of the grit hopper and 40 feet northeast of the fire hydrant.

Table 4 provides the boring number, sample interval, soil description, and analytical parameters for samples collected from Boring No. 16.

Table 5 provides the analytical data for the parameters that tested above the detection limits.

Previous Sediment Investigations

Since 1992, five previous sediment investigations have been conducted at or near the PSY. Table 6 summarizes these investigations. In 1992 and 1994, the Port collected sediment samples after dredging the Dry Dock 4 and 3 basins, respectively. In 1997, the Corps of Engineers (COE) and Environmental Protection Agency (EPA) collected samples throughout the Portland Harbor, including near the PSY. The former investigation was in support of the COE's channel deepening project. The latter was in support of a Harbor-wide site assessment. In 1997 and 1998, Cascade General collected sediment samples as part of its proposed purchase of the PSY. Cascade General and the Port jointly completed a comprehensive assessment of PSY sediments in 1998, also as part of the proposed sale.

Dredging History

This discussion was taken from a December 9, 1999 letter from the Port to DEQ on historical dredging and dredged material disposal activities at the PSY. Drawings and aerial photographs substantiating the timing of activities and quantities of dredged materials were included with the letter to DEQ. Table 7 provides a more complete summary of historic dredging activities at the PSY.

Early Dredging Near Swan Island

The first major dredging that occurred in the vicinity of the PSY was in the 1920's when the Port initiated development of Swan Island. As was discussed earlier, the main channel of the Willamette River was moved from the east side of Swan Island to the west-side to provide ships access to the upper Portland Harbor. The dredged materials were placed on Swan Island.

Early Dredging at the PSY

Dredging may have been conducted in both 1942 and 1950 when the basins for Dry Dock 1 and Dry Dock 2, respectively, were constructed. The Port has not located records regarding the amounts of materials, if any, generated during these construction projects.

Shipway Abandonment

Sometime after 1948, the Port started to abandon five of the eight shipways that were constructed in 1942. Shipways 4,5,6, 7 and 8 were abandoned in place by filling them with dredged materials. The shipways were almost completely filled by 1957. Some of the dredged materials placed in the shipways may have come from the 1957 construction of the Dry Dock 3 basin. The Port has not located records regarding the amount of materials, if any, generated during this construction project. The quantity of dredged materials from Dry Dock 3 probably only represents a small portion of the total amount of materials placed in the shipways given the total estimated dredged material disposal capacity for the shipways was 650,000 cubic yards.

In 1961, approximately 1-foot of sediment was dredged from the bottom of the Berth A (i.e., Dry Dock 1) basin and approximately 6-feet of sediment was dredged to create a slough trench along the south-side of the basin. The estimated amount of sediment generated during this activity was 4,000 cubic yards (in-situ volume calculated based on the difference between pre-dredging and post-dredging river bottom elevations). The dredged materials were placed in-water to the west of abandoned Shipways 7 and 8. These materials may have been removed from this area in 1977 when the Dry Dock 4 basin was constructed. As will be discussed below, dredged materials generated during construction of the Dry Dock 4 basin were placed at the end of Swan Island Lagoon.

In 1962, approximately 400 cubic yards of sediments were dredged from the Berth C (i.e., Dry Dock 3) basin and placed in the abandoned shipways.

Berth Maintenance Dredging

In 1962, sediments along Berths 306 through 308 were dredged to a depth of –20 feet (presumably referenced to Columbia River Datum, "CRD"). The estimated volume of sediment generated by this dredging activity was 3,000 cubic yards. The dredged materials were disposed in-water in the Swan Island Lagoon directly across from Berth 307.

Filling of Mocks Bottom

No PSY maintenance dredging materials were placed in Mocks Bottom during 1961 and 1962 as was stated in DEQ's File Review Memorandum. Rather, the dredged materials that were placed in Mocks Bottom initially came from the 1966 dredging of the Willamette River channel northwest of Swan Island, dredging of the Swan Island Lagoon, and dredging of the navigation channel south of Swan Island. By 1966, the filling of Mocks Bottom had occurred north of the Union Pacific Railroad tracks.

Aerial photographs indicate that the filling of Mocks Bottom occurred over period of approximately 15 to 18 years. In 1980, there was little to no development of the Mock Bottom area, while in 1984 several buildings and new roads (the extension of N. Leverman Street and the eastern portion of N. Cutter Circle) were present on the east-side of Mocks Bottom. It should be noted that significant quantities of dredged materials were ultimately placed in Mocks Bottom as part of a land reclamation program. The estimated dredged material disposal capacity of Mocks Bottom (Area C) was 5,300,000 cubic yards. Given the distance from the shoreline to Mocks Bottom and the need for structural fill, these materials were hydraulically dredged and piped to Mocks Bottom during multiple borrow-dredging operations (to obtain sand), as opposed to sediment from maintenance dredging which would have been mechanically dredged and disposed of at barge-accessible sites.

Filling of Swan Island Lagoon

The next PSY maintenance dredging activity was the 1977 construction of the Dry Dock 4 basin. Approximately 34,000 cubic yards of dredged material from this construction project may have been placed at the end of Swan Island Lagoon, specifically the east end of the Lagoon, which at the time was open almost all of the way to the intersection of N. Basin Avenue and N. Going Street. Soundings conducted in the portion of the Lagoon designated for disposal of Dry Dock 4 dredged materials indicate that the materials may have been placed starting at an elevation of between –22 feet and –4 feet (City of Portland Bench Mark No. 2071 used as a datum).

The disposal of dredged materials at the end of Swan Island Lagoon was consistent with the 1973 Lower Willamette River Management Plan prepared by the Oregon Division of State Lands. According to this Plan, filling of the Lagoon with channel maintenance dredging materials was permitted. Maintenance dredging materials were defined in the Plan as the materials needed to maintain the existing navigation channel and

access to water dependant facilities. According to the Lower Willamette River Management Plan, filling of the end of the Lagoon was limited to the eastern property line of Progress Electronics (located immediately east of Building 70 and Berth 308 on N. Lagoon Avenue) to a height of +32 feet CRD.

Parties other than the Port also placed dredged materials at the end of the Lagoon. Some of the additional fill materials came from hydraulic maintenance dredging of the Willamette River navigation channel. The Port intends to identify other parties who placed dredged materials at the end of the Lagoon.

In 1981, maintenance dredging was conducted at Dry Dock 3. Approximately 6 to 7 feet of sediment was removed from the bottom of the basin. The estimated volume of dredged materials was 7,000 cubic yards (in-situ volume calculated based on pre-dredge soundings and the final design dredge depth of –54 feet CRD). The dredged materials were placed at the end of Swan Island Lagoon, west of the current location of N. Anchor Street.

In 1985, Berths 301 through 305 were dredged to a design depth of – 33 feet CRD. Port records indicate that 23,667 cubic yards of material were taken to the end of the Lagoon. The dredged materials were placed at an elevation of approximately +4 feet CRD.

Also in 1985, the Port constructed Berth 315. This construction project included the dredging of 153,000 cubic yards of Willamette River sediments. Berth 315 is located upstream from the primary operating areas at the PSY (i.e., dry docks and Swan Island berths). These dredged materials were also placed at the end of Swan Island Lagoon.

By the mid- to late-1980's, most of the end of Swan Island Lagoon was filled. The Lagoon dredge fill plan indicates that navigation channel materials were hydraulically dredged from two borrow areas, one just downstream of the Fremont Bridge and one near Terminal 1. These dredged materials were piped to Swan Island Lagoon and used to fill the area west of N. Anchor Street. The elevation of the top of the fill was +26 feet CRD. By 1989, the fill was completed.

Based on the Port's records, it is estimated that approximately 218,000 cubic yards of PSY dredged materials were placed in the end of Swan Island Lagoon. Approximately 70 percent this material (i.e., 153,000 cubic yards) came from the construction of Berth 315, rather than from maintenance dredging near the primary PSY operating areas. The remaining materials did come from the primary operating areas. These materials were placed on or near the bottom of the original Lagoon. Dry Dock 4 and possibly Dry Dock 3 dredged materials are covered by over 30 feet of dredged materials from other locations, including the main channel of the Willamette River. The Berth 301 through 305 dredged materials are covered by approximately 20 feet of other dredged materials. The portion of the total dredged materials at the end of Swan Island Lagoon that came from the PSY is small compared to the 4,850,000 cubic yard estimated dredge material disposal capacity for this area.

Confined, In-Water Disposal at Ross Island

After 1985, there were three, not five, other dredging activities conducted at the PSY. The 1992 Berth 311 and 1995 Berth 309, 310 and 311 dredging actions listed in Table 1 of DEQ's File Review Memorandum were planned, but never occurred. The three that did occur were the 1992 dredging of 78,000 cubic yards of sediment from the Dry Dock 4 basin, the 1992 dredging of 17,000 cubic yards of sediment from the Dry Dock 1 basin, and the 1994 dredging of 21,000 cubic yards of sediment from Dry Dock 3. Dredged materials for all three of these projects were permitted by the COE, Division of State Lands, and DEQ and accepted by EPA for confined in-water disposal and placed in the contained, in-water disposal area at Ross Island.

Previous Remediation Activities

Previous Upland Remedial Actions

Since the late 1980's, a number of upland remedial actions have been conducted at the PSY. These remedial actions are summarized in Table 8. Most of the remedial actions conducted to date were related to the removal of USTs and HOTs, soils containing TPH and PCBs in the BWTP area, and sandblast grit and yard sweepings from the N. Channel Avenue Fabrication Site.

PRELIMINARY CONCEPTUAL SITE MODEL

Figures 4 and 5 illustrate the preliminary conceptual model for human and ecological receptors, respectively, at the PSY.

Land Use

The Site consists of 94 acres of uplands and 106 acres of submerged lands. Land use at the Site is industrial. The Site is zoned IH (industrial). The land use of surrounding properties is also industrial. The Site is surrounded on three sides by ODSL-owned submerged lands.

Local Geology

The Site is underlain by fill, fine sand, and silt. Fill materials are largely dredged materials. A portion of these dredged materials came from the Willamette River starting in 1923 when the Port relocated the main navigation channel to the west side of Swan Island and raised Swan Island to an elevation of 32 feet above mean low water. Dredged materials were placed on the north end of the Site when the shipways were abandoned in the 1950s when the Port constructed Dry Dock 2 and Dry Dock 3 basins.

Underlying the fill materials are fine sand and silt alluvium deposited by the Willamette River to form Swan Island. Figure 6 shows a generalized north-south subsurface cross section for the Site. To the north is Swan Island Lagoon. To the south is the Willamette River.

Beneath the sandy fill alluvium is the Troutdale Formation from approximately 100 to 400 ft bgs.

Local Hydrogeology

Groundwater Occurrence

The depth to groundwater at the Site is between 17 and 21 feet bgs, based on sampling conducted in May of 1998. Daily and seasonal variations in the depth to groundwater occur in response to variations in precipitation and river elevations.

Groundwater Gradients

There are two dominant components of long-term, net groundwater flow at the Site. One component is parallel to and in the same direction as

streamflow in the Willamette River. The second component is laterally outward from the center of Swan Island to the Willamette River and Lagoon. Figure 6 illustrates second component of groundwater flow.

Hydraulic Conductivities and Groundwater Flow Velocities

The hydraulic conductivity of aquifer materials and rate of groundwater movement underlying the PSY have not been estimated.

Local Hydrology

The Site is surrounded on three sides by surface water. Swan Island Lagoon borders the north side of the Site. The Willamette River borders the west and south sides of the Site. There are no other surface water bodies on or immediately adjacent to the Site.

Based on streamflow measurements made by the United States Geological Survey (USGS) at their Portland gauging station, located at river mile 12 approximately 3 miles upstream of the PSY, the average annual discharge for the Willamette River is 33,000 cubic feet per second. Streamflow exceeds 100,000 cubic feet per second approximately 5 percent of the time. Although the river discharge is relatively large, water velocities in the vicinity of the PSY are low. During a 1994 USGS study of sediment oxygen demand in the lower Willamette River found that maximum water velocities reached 0.5 feet per second during both rising and falling tides (Caldwell and Doyle, 1995). During a 1992 study of the effects of waterway development on anadromous and resident fish, water velocities in the Portland Harbor were found to range from 0.16 to 0.56 feet per second, with an average of 0.33 feet per second at a flowrate of 62,200 cubic feet per second (Port of Portland, 1992).

River elevations in the Portland Harbor fluctuate on a daily basis in response to tidal fluctuations in the Columbia River. These tidal fluctuations result in a reversal of water movement in the lower portion of the Portland Harbor. During times of medium to low flow in the Willamette River, tidal effects extend well upstream of the PSY.

Based on the PSY sediment investigation conducted in1998 (Striplin, 1998), river bottom sediments and the benthic community have different characteristics. There are four distinct regimes:

- The Willamette River side of the PSY where sediments are coarse-grained, there is some physical disturbance of near-surface sediments, and there are surface dwelling benthos. These conditions are consistent with the fact that river currents are higher in the main river channel and, therefore, there is less opportunity for deposition of fine sediments.
- The dry dock area where sediments consist of very fine sands, there is considerable physical disturbance of near-surface sediments, and there are surface dwelling benthos. These conditions are consistent with the fact that river currents are expected to be lower in this area

allowing for the deposition of finer sediments, and that these sediments are highly disturbed by ship traffic and the movement of the dry docks.

- The Lagoon where sediments are soft and unconsolidated, there is limited evidence of physical disturbance, and the benthos are surface dwelling. These conditions are consistent with the fact that there is negligible flow through the Lagoon allowing for the accumulation of finer sediments. Boat and ship traffic through the Lagoon likely keep some finer sediments in suspension creating soft, unconsolidated bottom conditions.
- Downstream of the PSY where sediments consist of layers of very fine sands and silts, there is low physical disturbance, and there are deep-dwelling benthos. This area is physically stable because river currents are low and the impacts of boat and ship traffic are limited because the water is deep.

Contaminant Source, Transport, and Fate

Source Areas

Known sources at the PSY are associated with ship hull and parts surface preparation and painting, ballast water treatment, petroleum storage, and fabrication and steam cleaning operations. Specific known sources include:

- Past ship hull washing, abrasive blasting and painting in dry docks
- Past ship hull abrasive blasting and outfitting at Berths 302, 303, 304, 305, 312, 313, 314, and 315
- Past ship parts surface preparation and painting in the Paint Shed/Blast Booth area
- Historic fabrication and painting of modules and petroleum storage in the N. Channel Avenue Fabrication Site
- Past Ballast Water Treatment Plant (BWTP) operations
- Fabrication and steam cleaning activities near Buildings 43, 50 and 80

Suspected sources include:

- Ship parts surface preparation and painting in Building 73
- Hazardous waste storage near and fabrication/manufacturing activities in Building 4
- Hazardous waste storage in the West States Incorporated (WSI) Storage Area
- PCB-containing electrical equipment formerly present in substations
- Condensate discharges from the old boiler

Petroleum storage in underground storage tanks (USTs) and heating oil tanks (HOTs) is not considered to be a source. Most of the USTs and HOTs have been removed from the Site. No Further Action (NFAs) determinations have been obtained from DEQ for all but one of the tanks where releases have been discovered. The Port is in the process of submitting a final cleanup report for this UST. There are only four active tanks at the PSY. Two of them are double-walled, fiberglass USTs with interstitial monitors. The other two are HOTs located in a concrete vault that were recently found to be tight when leak tested.

Releases from these known and suspected sources are largely historical. The Port and Cascade General have implemented a wide array of pollution controls and management practices to significantly reduce current releases from the sources, including:

- **Ballast Water Treatment Plant** – The BWTP accepts oily waste from vessel cleaning and maintenance. Oil is extracted by natural separation, addition of chemicals, and heating the wastewater. The reclaimed oil is recycled. Treated water is tested before it is either pumped to the city sewer system or discharged to the Willamette River under NPDES permit. Thus, currently there is control over the quality of any water discharged from the BWTP.
- **Storm water Treatment System** – The storm water treatment system is used to treat water generated by hull washing and other activities conducted on the dry docks. This system was placed into operation in 1997 and has significantly reduced current releases associated with washing and cleaning of ship hulls.
- **Dry dock operation** – As of 1993, the dry docks are completely cleaned before they are lowered to prevent the release of sand blast grit and other solid materials into the Willamette River. Cleaning of the dry docks consists of removing all loose debris, equipment, and materials, followed by sweeping to remove ABM. Each dock is visually inspected before it is lowered into the river. Curtains are placed around each dry dock, while in use, to prevent airborne dust and debris from entering the river.
- **Yard Sweeping** – The paved portions of the PSY are swept on a regular basis to prevent materials from entering the storm water collection and management system.

Chemicals of Interest

Upland

Based on the previous upland site investigation studies and remedial actions conducted at the PSY, the chemicals of interest (COIs) are metals, volatile organic compounds (VOCs), TPH, polynuclear aromatic

hydrocarbons (PAHs), and PCBs. Table 9 summarizes the COIs for each of the known and suspected sources.

Sediment

Biological tests conducted near the PSY are consistent with the bulk chemistry results in that they indicate that the extent of sediment contamination is limited. Of the 52 bioassay tests that were conducted as part of the 1998 PSY sediment investigation, 34 were conducted on sediment samples collected near the PSY, including Berth 311. The bioassay results can be summarized as follows:

- None of the 34 chironomid bioassays conducted near the PSY indicated the potential for adverse effects. Only 9 percent (3 of the 34) amphipod bioassays conducted near the PSY had survival rates that indicated the potential for adverse effects. Only 14 percent (5 of the 34) Microtox™ bioassays conducted near the PSY indicated the potential for toxiticity.
- There was only one station (in the small boat basin) where two bioassay hits occurred indicating the potential for adverse effects.
- With the exception of one station on the Willamette River side of the PSY and one station downstream of the PSY, all of the stations where bioassay results indicate a potential for adverse effects are located adjacent to the shoreline.

Preliminary Apparent Effects Thresholds (AETs) were calculated for PSY sediments based on the benthic bioassay results. The following COIs were identified based on a comparison of the AET values and the sediment chemistry.

- **Willamette River side of the PSY** - Sediments principally contained low molecular weight polynuclear aromatic hydrocarbons (LPAHs), high molecular weight polynuclear aromatic hydrocarbons (HPAHs), and phthalates potentially associated with historic BWTP operations and ship hull surface preparation and painting activities conducted in Dry Dock 4. The constituents present in this area were largely confined to sediment near Berths 312 and 313.
- **Dry dock area** - Sediments principally contained metals associated with ship hull surface preparation and painting, such as copper, zinc, and nickel. To a lesser extent, organics such as HPAHs, LPAHs, and phthalates were present. In most locations, these metals and organics were present in near-surface sediments immediately adjacent to the dry docks. The exception to this trend was in each of the dry dock basins where constituent concentrations tended to be higher at depth. "Cleaner" sediments appear to overly these sediments indicating that recent source control measures implemented at the dry docks have been effective in reducing releases to the river.

- **PSY side of the Lagoon** - Fewer constituents associated with ship hull surface preparation and painting were present. The constituents that were present tended to vary with location. At Berth 302, PCBs are the principal chemical present in sediments. The likely potential source of the PCBs was one the substations located near Berth 302. Zinc, dimethylphthalate, and LPAHs were present at Berths 303 through 305. These constituents likely originated from ship outfitting activities conducted at these berths. Mercury was present near Berths 306 and 308. The potential source of the mercury is unknown. The available sediment chemistry data indicate that these constituents were largely present in the immediate vicinity of the berths.
- **Tributyltin** - One chemical that is present in several of the areas discussed above is tributyltin (TBT). TBT is primarily present in the dry dock area, which is consistent with its likely historic sources of hull washing, abrasive blasting, and painting. TBT was also present in lower concentrations in the Lagoon and on the Willamette River side of the PSY, potentially from historic ablative losses of TBT from ship hulls while in transit to and from the berths. Generally, TBT concentrations decline rapidly with distance from the dry docks indicating limited transport.

Transport and Fate

Figures 4 and 5 illustrate the potential contaminant migration and exposure pathways for the Site. The principal contaminant migration pathways include:

- Air- and water runoff-related transport to the Willamette River and Swan Island Lagoon from historic ship repair and outfitting activities conducted at the dry docks and berths
- Runoff-related transport from historic and current upland sources like electrical substations, module fabrication and painting, and ship parts surface preparation and painting
- Historic direct discharges to the Willamette River from the BWTP and, possibly, the old boiler
- Potential subsurface transport of constituents beneath the former BWTP ponds
- Wind erosion of surface soils containing constituents from historic and current sources like module fabrication and ship parts surface preparation and painting
- Volatilization into air from soils containing VOCs; this may not be a relevant pathway at the Site given that few VOCs have been detected in soil at the Site.
- Leaching from soil to groundwater; this may not be a relevant pathway, except potentially at the BWTP, because previous upland site investigation results indicate that COIs are present mainly in

surface soils (i.e., upper 2 to 3 feet). Even at Building 58 where TPH was left in place above Level 2 UST cleanup levels, groundwater was not impacted and DEQ recommended no further action. The leaching from soil to groundwater pathway may also not be a relevant pathway because the COIs detected to date are relatively immobile. Finally, the leaching from soil to groundwater pathway may not be a relevant pathway because the site is largely paved or covered with buildings.

Exposure Pathways

Potential human exposure pathways for the Site include uptake of constituents by fish, incidental ingestion and dermal contact with surface water, incidental ingestion and dermal contact with surface soil, and inhalation of particulates.

Potential ecological exposure pathways for the Site include direct uptake from sediment and water.

SITE CHARACTERIZATION PLAN

Data Gaps

In its File Review Memorandum, DEQ identified a specific set of data gaps that could either be addressed through supplemental information on past operations and environmental conditions or through site characterization. This section discusses each of these data gaps and the need for additional site characterization work.

Upland

Former Ballast Water Treatment Plant

The original BWTP was constructed in 1971 to separate contaminants, primarily petroleum products, from ship ballast water. The BWTP facilities plan for 1971 shows that the following structures were part of the plant: asphalt-bottom, sealed 60,000 barrel holding pond; asphalt-bottom, sealed 10,000 treatment pond; three 22,400-gallon aboveground oil storage tanks; 6,000 gpm primary separator; and a 300 gpm treatment channel and secondary separator. In 1972, four additional holding ponds were constructed east of the original holding and treatment ponds. The four additional holding ponds were likely lined with asphalt, just like the original holding and treatment ponds. The four holding ponds were active until 1979. Filling of the ponds occurred between 1979 and 1981. Building 72 was constructed on top of the four former ponds (see Figure 7).

Prior to the construction of the BWTP, the area was used to store wood and other miscellaneous debris from 1967 to 1971. Prior to 1967, the area was being filled by the Port during shipway abandonment.

Between November 1992 and March 1993, the Port sampled soils in two areas within the BWTP: Tank 10 and the Pipe Area (Hahn and Associates, 1993). TPH and PCBs were detected near Tank 10 at concentrations ranging from 10,000 to 59,000 mg/kg and 0.78 to 1.2 mg/kg, respectively. One sample from the Tank 10 area was analyzed for VOC and TCLP metals. No VOCs were detected and no TCLP metals were detected, other than barium that was present in the extract at a concentration of 0.96 mg/L. TPH was also detected in the Pipe Area at concentrations ranging from 84 to 27,000 mg/kg; PCBs were not analyzed in this area.

Based on these results, the Port decided to remove soils in both areas. Approximately 20 tons or 13 CY of soil were removed from the area around Tank 10. On the east side of the tank and on a portion of the south side of the tank, soils were removed to a depth of 0.5 feet before

encountering concrete. On the remainder of the south side and on the west side of Tank 10, the depth of excavation ranged from 3 to 6 feet bgs. Table 10 summarizes the confirmation sampling results. The locations of the Tank 10 confirmation samples are shown in Figure 8.

Approximately 46 tons of 31 CY of soil were removed from the Pipe Area by excavating soils to a depth of 2 to 4 feet bgs. Table 10 summarizes the confirmation sampling results. The locations of the Pipe Area confirmation samples are shown in Figure 8.

Also in 1992, the Port conducted an assessment of the soils in the vicinity of four 84,000-gallon aboveground storage tanks at the BTWP. Figure 8 shows the location of the four tanks. The investigation was undertaken to determine if previous activities related to the operation of the aboveground storage tanks may have resulted in petroleum product leaks or spills (Hahn and Associates, 1992a). Soil samples were collected at the surface (i.e., 6-inches below ground surface, bgs) and 2.5- to 3-feet bgs. The eight soil samples were analyzed for TPH using EPA Method 418.1. TPH concentrations were found to range from 170 to 2,200 mg/kg in the surface samples and 120 to 1,100 mg/kg at a depth of 2.5 to 3 feet.

In late 1993 and early 1994, the Port removed soils beneath the four tanks prior to the installation of a concrete secondary containment system (Hahn and Associates, 1994). A minimum of 1-foot of soil was removed from most of the area underlying the tanks; in several locations soils were removed to a depth 3 to 5 feet. A total of 69 tons or approximately 46 CY of soil were removed. Table 11 summarizes the confirmation sampling results for TPH and PCBs. Figure 9 shows the confirmation sample locations.

In 1998, the Port and Cascade General jointly completed one boring (i.e., Boring 9) in the BWTP area. Boring No. 9 was located in the middle of the access road 14 feet west of the tank farm containment wall and 45 feet south of the end of the access road. Table 12 provides the boring number, sample interval, soil description, and analytical parameters for samples collected in the BWTP area.

Table 13 provides the analytical data for the parameters that tested above the detection limits.

Based on the soil samples collected to date and the historical operations conducted in the BWTP area, additional investigation will be conducted as part of the RI. The scope of the investigation sampling is described later in this section.

N. Channel Avenue Fabrication Site

The N. Channel Avenue Fabrication Site is located in the southeast portion of the Site between N. Channel Avenue and the Willamette River (see Figure 2). It is currently used for outdoor storage of equipment, steel, cable drums, empty portable tanks and totes, and other materials. Wood waste recycling also occurs in this area. Building 83 and the area immediately around Building 83 was used for storage and parking of trucks.

From 1986 to 1990, the area was used by the Atlantic Richfield Company (ARCO) for the construction of modular units used for oil processing on Alaska's North Slope. Eight 2,700-ton modules were constructed in 1986, and seven 3,400-ton modules were constructed in 1990. Fabrication, finish painting and the application of fire retardant were conducted on concrete pads in the center of the area, with material storage, administrative modular trailers, and equipment stored around the perimeter of the area. A portable fire safety shed was constructed on the west side of the area. The shed is still present and used as the Shipyard University. Building 83 was constructed as part of the ARCO modular fabrication project. This building served as a general shop and vehicle maintenance repair area. The building has a wood-frame construction on a concrete slab floor.

In 1978, the area was used as the staging and pre-cast concrete construction site for the BWTP.

Between 1978 and the early 1940's the N. Channel Avenue Fabrication Site was primarily open, graded soil with railroad spurs used for material receiving and storage. A salvage building was located in the west-central portion of the area.

Petroleum, fuel and solvents were stored in tanks and totes during the ARCO construction project. Three ASTs were located along the south side of Building 83.

In December of 1989, the Port conducted an Environmental Review (ER) of the N. Channel Avenue Fabrication site (Hahn and Associates, 1989). At the time of the ER, permanent improvements included Building 81, an office building, and Building 83, a mechanical shop used for equipment maintenance. The following summarizes key findings of the 1989 ER:

- There were no visual indications of oil spills associated with the storage and use of oil to test gas turbines installed on some of the modules
- Visible staining of soil was observed in the vicinity of an oil storage shed located south of Building 83 (see Figure 10). This shed was used to store drums of petroleum products (motor oil, lubricants, and greases)
- Apparent petroleum staining of the gravel surface east of Building 83 was observed (see Figure 10)
- Gasoline and diesel fuel were being stored in two 500 gallon ASTs located south of Building 83; gasoline was being stored in a portable truck-mounted tank in the same area
- There were no visible signs of paint or paint thinner stains in or around the roll-off box used to store these materials and a locker where paint brushes were cleaned on the north side of the N. Channel Avenue Fabrication Site
- There were no visible signs of photographic chemical spills near a portable trailer used for non-destructive X-ray examinations of welds

located on the northern side of the N. Channel Avenue Fabrication Site

- Small losses of a 60 percent solution of ethylene glycol in water may have occurred in various locations when the solution was used to hydrotest piping systems installed in the modules.
- Two transformers were located on the northern side of the area, one was a non-PCB transformer and the other contained less than 50 ppm PCBs
- There was no visual evidence of USTs

A subsequent ER was performed on July 20, 1990 (Hahn and Associates, 1990). This ER was performed at a time when the area was largely vacant, the oil production modules had been loaded, and temporary structures dismantled. The following additional observations were made:

- There was an area of visual staining near the fabrication shop on the west end of the N. Channel Avenue Fabrication Site (see Figure 10)
- Sandblast sand was observed on the ground on the west side of the N. Channel Avenue Fabrication Site (see Figure 10)

In January of 1990, the Port conducted an investigation in the vicinity of the oil storage shed where during the 1989 ER stained soils were observed. An attempt was made to complete six hand auger borings (see Figure 10). At each boring location, a buried concrete pad was encountered at a depth of approximately one foot. Based on the presence of the pad, the Port decided not to analyze any soil samples.

In 1993, 60 cubic yards of sandblast sand and cadmium-contaminated yard sweepings were removed from the area where they were observed during the 1990 ER. These materials were characterized as hazardous based on cadmium levels and disposed of at a hazardous waste landfill. The Port collected eight, 0- to 0.5-foot bgs verification soil samples from the removal area. The samples were collected using approximately a 12- by 12-foot, systematic, random sampling grid. All eight samples were analyzed for TCLP metals. Cadmium was the only metal detected in the TCLP extract; it was detected in five of the eight TCLP analyses at concentrations ranging from 0.028 to 0.13 mg/L (Hahn and Associates, 1995).

In 1998, the Port and Cascade General jointly completed five borings in the N. Channel Avenue Fabrication Site. The five sample locations were selected based on historical and current site activities. Geoprobe penetrations were completed to the water table at Borings No. 2, 3 , 4 , 5, and 6 (see Figure 10).

Boring No. 2 was located in an area of observed soil staining approximately 55 feet west of the southwest corner of Building 83.

Boring No. 3 was located approximately 130 feet southwest of Building 83.

Boring No. 4 was located 255 feet southwest of Building 83.

Boring No. 5 was located approximately 400 feet east of Building 81 and 200 feet south of the fence along North Channel Avenue.

Boring No. 6 was located 175 feet south of Boring No. 5.

Table 14 provides the boring number, sample interval, soil description, and analytical parameters for samples collected in the fabrication site.

Table 15 provides the analytical data for the parameters that tested above the detection limits.

Based on the soil samples collected to date and historical activities conducted in the N. Channel Avenue Fabrication Site, additional investigation will be conducted as part of the RI. The scope of the investigation is described later in this section.

Building 73

Sandblasting, grit blasting, metal shot blasting, and parts painting are conducted in Building 73. The interior of the building is divided by walls for each functional area. The south half of the building is used for surface preparation and north half is the paint booth.

Building 73 was constructed in 1980 and 1981. Prior to its construction, the area was used for lay-down of shipbuilding parts during the Kaiser shipbuilding era and as an unpaved storage yard between during the 1950s, 1960s and 1970s.

Chemicals used at Building 73 include solvents and oil-based paints. Blasting media are also stored and used in the building. Solvents are stored in dip tanks located on the south wall of the building. An environmental audit conducted in 1998 found that the dip tanks were in good condition and there were no visible leaks or spills. Waste solvents are placed in drums for pick-up by the central waste collection group or distilled onsite. Waste paint storage also occurs in a temporary hazardous waste storage area located on the south side of Building 73, in an outdoor, sheltered area and inside intermodal containers. A solvent distiller is located inside an intermodal container adjacent to Building 73. The refined product is reused and the concentrated still bottoms are placed in a 55-gallon drum and moved to the waste paint storage area. Waste paints are disposed offsite as a hazardous waste. During the 1998 environmental audit, there were no visible indications of paint spills or releases inside the building.

Blasting media are carried out of the east-end of the building as fugitive dusts. A sweeper is used to capture blasting media that deposits on the pavement.

To date, no site investigation or remediation has been conducted at Building 73.

Based on the historical activities conducted at Building 73, investigation will be conducted as part of the RI. The scope of the investigation is described later in this section.

Building 4

Building 4 is the largest building at the PSY. It is located in the central portion of the Site. The building is divided into 11 parallel bays, with a paved parking and storage area at the west and east end of the building. The floor is concrete and asphalt. The building was built during 1942.

During the Kaiser shipbuilding era, Building 4 was used for fabrication, primarily ship assembly and metal working. After 1949, Building 4 continued to be used for fabrication. Several of the bays were also leased to tenants for use store surplus property, manufacture heavy equipment, store steel, store grain, and other varied uses.

Petroleum products as fuels and lubricants and solvents were used and stored at Building 4. Since the early 1990s, the Port has performed regular inspections of leased bays as tenants vacate their areas. As a result of those inspections, the Port has identified and systematically disposed of wastes and conducted cleanup actions, such as steam-cleaning stained floors. Hydraulic systems for the large roller and press in the eastern end of Bay 10 were observed to be leaking into concrete reservoirs in the floor. Staining of the concrete beneath this equipment was observed during a 1998 environmental audit. Cascade General sampled the oil in this equipment in 1998 and found that it did not contain PCBs.

Some of the bays have a floor drain that historically collected roof and floor runoff. These floor drains were connected to the storm water system. In 1994 or 1995, the floor drains were sealed. Figure 11 shows the approximate location of the suspected storm sewer that previously received discharges from the floor drains. Additional records review and site reconnaissance are required to locate the Building 4 floor drains and confirm the storm sewer location.

Between 1985 and 1994, WSI may have stored drums containing waste materials on the northeast side of Building 4, near Bays 2 through 5.

Based on the historical activities conducted at Building 4, additional investigation will be conducted as part of the RI. The scope of the investigation is described later in this section.

Underground Storage Tanks (USTs) and Underground Heating Oil Tanks (HOTs)

Eleven USTs were removed at the PSY between October of 1989 and March of 1992. Table 16 summarizes the status of the 11 former USTs and Figure 12 shows where these USTs were located. Table 8 lists the quantities of soil removed during UST decommissioning.

No Further Action (NFA) determinations have been received from the DEQ on 6 of the 11 USTs. Groundwater monitoring was performed at two of the USTs and they are now closed. No contamination was found at the other three USTs when they were removed.

Currently there area four active USTs at the PSY. Two of the USTs, No. 15 (PSY-1A) and No. 16 (PSY2A), are 6,000 gallon gasoline and diesel

storage USTs located in the card lock fueling area. These two tanks were installed in 1989 and are constructed of dual-walled fiberglass with interstitial monitors. Two of the USTs are 20,000 gallon heating oil storage tanks (HOTs) located at the Central Utilities Building (CUB). These two tanks are single-walled steel tanks in a concrete vault. The CUB tanks were tested in 1998 and were found to be tight.

In January of 1991, the Port conducted an investigation, at the request of DEQ, to define the extent of petroleum hydrocarbon contamination at a previously decommissioned UST No. 9 located east of Building 58. Soil samples were collected from seven soil borings. Three of the borings were completed as monitoring wells; these wells were later abandoned by the Port. The soil sampling results found that petroleum hydrocarbons were present at concentrations between 2,800 and 48,000 mg/kg, primarily at depths of 12 to 17 feet. The petroleum hydrocarbons were found to be diesel fuel and a heavier petroleum product, possibly boiler fuel (Hahn and Associates, 1991). Additional investigation to further define the extent of petroleum hydrocarbons was limited by the presence of structures and operating electrical transformers. Analytical testing of groundwater samples collected from the monitoring wells found that benzene, ethylbenzene, toluene and xylenes (BETX) were not detected during four quarters of sampling at a detection limits ranging from 0.5 to 1 ug/L (Hahn and Associates, 1991). Heavier petroleum hydrocarbons were detected at 0.7 to 0.9 mg/L during the first quarter of groundwater sampling. By the fourth quarter, no heavy hydrocarbons were detected in groundwater, at a detection limit of 0.5 mg/L (Hahn and Associates, 1991). Table 17 summarizes the soil sampling results and Figure 13 shows the location of the soil borings and former monitoring wells.

In 1992, the Port decommissioned a 1,200 gallon UST filled with virgin glycol to supply compressors in the CUB (Hahn and Associates, 1992c). According the Hahn and Associates, the Port's consultant, the UST was never connected to the compressors and its contents were never used. In accordance with a DEQ-approved plan, the tank and soil beneath the tank were visually inspected. No visual indications of holes or breaches in the UST were observed. In addition, no discolored soils or odors were observed.

In 1993, a release of heating oil was discovered in the concrete vault containing the two, 20,000-gallon HOTs. This release was reported to DEQ on April 20, 1993. Two soil investigations were conducted in 1993, and in early 1994 most of the soil containing petroleum hydrocarbons was removed and a concrete floor was poured in the bottom of the vault. In 1994, the Port conducted a site investigation to investigate potential groundwater impacts from the heating oil release (Century West, 1994a). Three soil borings were completed to a depth of 27.5 feet and two more soil borings were completed to a depth of 15 feet near the westernmost HOT. Temporary well points were installed in the three deep borings for purposes of sampling groundwater. Low to nondetectable levels of TPH (i.e., less than 9 mg/kg) were detected in soil samples collected between 15 and 27 feet bgs. No BTEX was detected, at detection limits of 2 ug/L, and no TPH was detected, at detection limits of 0.5 ug/L, in groundwater.

In 1997, two soil borings were completed for purposes of collecting soil samples for constituent analysis. These sampling results were used to evaluate the potential for a risk-based closure for the residual petroleum-contaminated soils. The Port proposed a risk-based closure in 1998 and DEQ rejected the Port's proposal. The Port subsequently removed 24.45 tons or approximately 16 CY of soil. Ten soil samples were collected from the excavation and six were analyzed for TPH and PAHs. The Port is in the process of evaluating the sampling results and preparing a revised risk-based closure proposal for the CUB HOTs.

Thus, all of the former USTs have been addressed. The only HOT issue that has not been addressed is the heating oil release near the CUB. A report documenting the investigation and cleanup of soils at the CUB and the Port's revised risk-based closure proposal is being prepared for submittal to DEQ.

The currently active USTs at the PSY are either new fiberglass tanks with interstitial leak detection, and the currently active HOTs are enclosed in a concrete vault and passed leak tests in 1998.

Thus, no additional investigation is planned for USTs and HOTs as part of the RI.

Paint Shed/Blast Booth Area

The paint shed and blast booths are located approximately 150 feet east of Building 73 and 200 feet west of Building 4. This area also includes the historic storage of sandblast grit near Berth 313.

In 1998, the Port and Cascade General jointly completed five borings in the paint shed/blast booth area: Boring Nos. 8, 10, 11, 12 and 13 (see Figure 14).

Boring No. 8 was located 180 feet south of Building 73, between Berth 313 and Berth 314. A historical aerial photograph dated 1981 on file at Cascade General Environmental Offices indicated that Berth 313 in the vicinity of Boring No. 8 was used as a grit blasting and grit storage area.

Boring No. 10 was located 175 feet west of Bay 7 of Building 4, on the north side of the paint shed.

Boring No. 11 was located 150 feet west of Boring No. 10.

Boring No. 12 was located 12 feet south of the compressed gas storage shed and 175 feet west of Bay 9 of Building 4. Boring No. 12 was completed in an area of visible soil staining.

Boring No. 13 was located 100 feet west of Boring No. 12.

Table 18 provides the boring number, sample interval, soil description, and analytical parameters for samples collected in the paint shed/blast booth area.

Table 19 provides the analytical data for the parameters that tested above the detection limits.

Based on the soil samples collected to date and the historical activities conducted in the Paint Shed/Blast Booth area, additional investigation will

be conducted as part of the RI. The scope of the investigation is described later in this section.

Building 43, 50 and 80 Area (Steam Cleaning Basin)

During the Kaiser shipbuilding era, the Building 43, 50 and 80 area was used for general lay-down and storage. Aerial photographs taken during this era indicate that this area was unpaved.

Building 43 was constructed in 1942 and used as a pipe fitting shop during the Kaiser shipbuilding era. Later, the first floor of the Building 43 was used for engine and boiler fabrication and repair. That use stopped sometime in 1998. A company that performs nondestructive testing of materials and weld inspections now occupies the second floor of the building.

Building 50 was constructed in 1951, with a 6,500-foot addition in 1962. It consisted of a metal working shop and administrative space. Currently, a company that specializes in marine propellers, occupies the eastern bays of the building. Their activities include cutting, welding, sandblasting, spray painting, hot dip cleaning (in a caustic solution), and steam cleaning. An abrasive blasting booth and spray booth are located within the building in Bay 3. A 1998 environmental audit found the concrete floor beneath the hot dip tank was in good condition and hazardous substance use was confined to the interior of the building. Steam cleaning is performed outside the northeast corner of the building. Wastewater from steam cleaning enters a drain with a grease trap that is reportedly connected to the sanitary sewer system. Cascade General occupies the western end, which includes a lunch area and restrooms. The entire second floor is used for administrative purposes. In the late 1960's, the Bay 4 was used for welding and Bay 3 was used as a machine shop; the remainder of the building was used for storage areas and dressing rooms.

From 1951 to 1993, Building 54 was located to the east of Building 50. The northern half of Building 54 was used for paint; oil and solvent storage. The company that now occupies Building 50 operates a steam-cleaning basin in this area. They also store large naval brass alloy ship screws and equipment in the parking lot east of Building 50.

Building 80 was constructed in 1944. Early on, it was used for ship outfitting. In 1947, the building's use was listed as ship dismantling. Consolidated Builders leased the building from 1949 to 1951. In the late 1960s, the building was the dry dock office and storage area. Currently, the building is used as office space and shop space. A portion of the shop space (southeast end of the building on the first floor) is used by a tenant for tank pumping/cleaning equipment maintenance and repair. The rest of the shop space is used by tenant for metal work, hydraulic repair, and turbine repair.

In August of 1992, the Port discovered soils containing petroleum hydrocarbons while re-surfacing a parking lot north of Building 50 (Hahn and Associates, 1992g). Soil samples collected in the area were found to

contain Stoddard solvent and diesel/oil petroleum hydrocarbons at concentrations up to 390 mg/kg and 1,500 mg/kg, respectively. PCBs were detected at concentrations of up to 0.09 mg/kg. Ethylbenzene and total xylenes were detected in one sample at concentrations of 0.68 mg/kg and 1.26 mg/kg, respectively. TCLP lead was detected at a concentration of up to 1.26 mg/L. Based on these results, the Port decided to remove 91 tons or approximately 61 CY of soils by excavating to a depth of up to 5 feet bgs. Two post-excavation soil verification samples were collected from the excavation sidewalls and one was collected from the floor of the excavation. Neither Stoddard solvent or diesel/oil petroleum hydrocarbons were detected in any of the verification samples, at detection limits of 3 mg/kg and 20 mg/kg, respectively. Figure 14 shows the location of the soil verification samples.

In 1998, the Port and Cascade General jointly completed a boring (i.e., Boring 14) in the area currently used as a steam cleaning basin (see Figure 15). This area was previously occupied by Building 54. Boring No. 14 was located in the fourth stall from the north end of the parking lot. Composite samples were collected from the surface to 2 feet bgs and from the 2-foot interval just above the groundwater level. Asphalt or gravel fill encountered on the soil surface was not included in the sample.

Table 20 provides the boring number, sample interval, soil description, and analytical parameters for samples collected in the area.

Table 21 provides the analytical data for the parameters that tested above the detection limits.

Based on the soil samples collected to date and the historical activities conducted in the Building 43, 50 and 80 Area (Steam Cleaning Basin), additional investigation will be conducted as part of the RI. The scope of the investigation is described later in this section.

Electrical Substations

There are a total of eight substations at the PSY (see Figure 16). Table 22 presents the date of construction, type of electrical equipment located in each substation, presence of PCBs in current electrical equipment, and condition of each substation. Since 1983, the electrical equipment in each substation has been inspected and surveyed for PCBs. A 1985 survey found that two transformers in Substation 1 and one transformer in Substation 4 contained PCBs. The one transformer in Substation 4 was removed from the Site in 1985. The two transformers in Substation 1 were removed from the Site in 1992. After that time, there were no PCBs in any of the in service transformers.

In 1992, Hahn and Associates sampled soil, concrete and electrical equipment in Substation 1 (Hahn and Associates, 1992b). Oil samples were collected from four transformers, six over-current breakers (OCBs), and one 55-gallon drum. Soil samples were collected from below one transformer, below two OCBs, from an unpaved electrical service trench, and from one soil depression. Wipe samples were collected from all 13 transformers, all 6 OCBs, 17 concrete pads, and 8 asphalt surfaces around Transformers 6 and 10. Insulating oils were found to contain

greater than 50 ppm PCBs. Soil was found to contain 0 to 4.1 mg/kg PCBs, and concrete contained greater than 100 ug/100 cm^2. Later in 1992 the Port decontaminated Substation 1 (Hahn and Associates, 1992d). The decontamination process was conducted in two phases. Phase I involved excavation of soil around Transformer 10, in one area along the boundary of Substation 1 west of Transformer 9, and along an electrical conduit ditch that ran to the east of Transformer 10 (Hahn and Associates, 1992d). Six wipe samples and five soil samples were collected for verification purposes. The verification wipe samples contained between less than 1 ug/100cm^2 and 9 ug/100 cm^2 PCBs. The soil samples contained between less than 0.05 mg/kg and 1.8 mg/kg PCBs. Phase II involved the removal Transformer 6 along with it associated concrete pad and asphalt surface within a 5 foot radius of the transformer (Hahn and Associates, 1992e). Three wipe verification samples, two soil verification samples, and three dielectric fluid samples were collected. The wipe samples contained between 3 and 8 ug/100 cm^2 of PCBs. The soil samples contained 0.06 and 0.74 mg/kg of PCBs. Dielectric fluid samples collected from Transformers 2, 3 and 4 contained 8, 27 and 3 mg/kg PCBs, respectively.

In 1998, the Port and Cascade General jointly completed a boring (i.e., Boring No. 15) near Substation 3 (see Figure 15). Boring No. 15 was located in the middle of the access road, approximately 35 feet east of Substation 3 and 80 feet south of Berth 302.

Table 23 provides the boring number, sample interval, soil description, and analytical parameters for samples collected from Boring No. 15.

Table 24 provides the analytical data for the parameters that tested above the detection limits.

Based on the known and suspected presence of PCBs in electrical equipment in the PSY substations, additional sampling will be conducted as part of the RI. The scope of the investigation is discussed later in this section.

Old Boiler

The original boiler for the PSY was located in Building 58. This building was constructed in 1957 as a diesel-fueled boiler house. The boiler provided steam heat to many of the buildings and berths on the north side of the PSY. Building 58 has two bays, is constructed of cinder blocks, and has a concrete floor. An addition was installed to the west-side of the existing boiler in the mid-1960s. The area around the building was paved when the building was constructed. The boiler operated until approximately 1979 when the new CUB was constructed. The building was converted to a garage-type structure after removing the boiler and USTs Nos. 9 and 10 in 1989 and 1990. Building 58 is currently used as a rigging shed and for storage of electrical cable, cables, tie down lines, and miscellaneous equipment.

A floor drain is present in the northernmost bay. It is possible that this drain received condensate discharges from the boiler. As will be discussed below, boiler blow-down was discharged to the Swan Island

Lagoon (at Berth 302) through former Outfall 002. It is also possible that the floor drain was connected to a recently discovered dry well located near the southwestern corner of the building.

Based on the historical operations conducted at the old boiler building, sampling will be conducted as part of the RI. The scope of the investigation is discussed later in this section.

Outfalls 002 and 003

DEQ's File Review Memorandum requested that information be provided on the status and environmental conditions of outfalls formerly named 002 and 003 in an old National Pollutant Elimination Discharge System (NPDES) Permit. According to Port files, prior to 1996 when the Port transferred the PSY NPDES permit to Cascade General, the "old" NPDES permit (File No. 70596) referenced three outfalls: 001, 002 and 003.

Former Outfall 001, the air compressor cooling water outfall, discharged at the head of Dry Dock 3. This outfall received cooling water from four compressors that were located in Building 60. The cooling water from these compressors discharged to floor drains connected to the outfall. As of 1989, the four compressors had been replaced with newer units that did not require a discharge permit. The compressors were not operated after 1986 and former Outfall 001 was not used after 1986. The floor drains in Building 60 have been plugged and former Outfall 001 likely remains in place.

Former Outfall 002, the boiler blow-down outfall, discharged to Swan Island at Berth 302. This outfall received blow-down from the steam boiler formerly located in Building 58. Discharge only occurred when the boiler was being cleaned to remove scale. When the new yard was constructed, the steam boiler was put on standby in 1986. As was discussed above, the boiler was removed from Building 58 in 1989 and 1990. Former Outfall 002 likely still remains in place, but is not in use.

Former Outfall 003, the treated ballast water outfall, discharges to the Willamette River at Berth 313. This outfall is still active and is referred to in the current NPDES permit (Permit No. 101393) as Outfall 001. It receives the discharge from tanks used to hold treated ballast water for testing prior to discharge.

Current Outfall 002, the treated dry dock storm water and process wastewater outfall, also discharges to the Willamette River at the same location as current Outfall 001.

No additional investigation is planned related to former Outfall 002. Sediment sampling was performed at Berth 302 during the 1998 PSY Sediment Investigation. Additional sampling is planned for the Dry Dock 3 basin, where former Outfall 001 discharged, and near the point of discharge for former Outfall 003 (current Outfall 001). The scope of the investigation is discussed later in this section.

Hazardous Waste Storage Areas

The current hazardous waste storage area is located near the east-end of Berth 305 (see Figure 17). The shed has a corrugated aluminum exterior siding on two sides, bermed concrete floors, and chain link fences. The hazardous waste storage area is surrounded by asphalt paving, which according to aerial photographs has been in since the mid-1950s. Hazardous waste storage started in this area in 1987. All hazardous wastes are stored in 55-gallon drums prior to being transported offsite. A 1998 environmental audit found that there were no significant visible stains or odors on the concrete in the current hazardous waste storage area.

Between approximately 1985 and 1994, hazardous wastes were also stored in a partially-paved and fenced area located southwest of Building 4 and north of Berth 313 (see Figure 2). This area is referred to as the WSI Storage Area. Drums of waste and cans of paint were stored in this area. In 1994, the WSI Storage Area was cleaned up under DEQ oversight. No sampling was conducted at the time.

In addition, some hazardous materials were stored north of Building 73 and near the southeast corner of Building 10 by the Norvac Company (Hahn and Associates, 1992f). A waste inventory conducted by the Port in 1991 and 1992 found that the Norvac Company was storing petroleum-contaminated debris and liquids in 55-gallon drums and aboveground storage tanks. A site investigation was conducted by the Port in September and October of 1992 after petroleum-contaminated soils were first detected during a limited investigation conducted in January and March of 1992. Soil samples were collected from six borings at depths of one, two, three and four feet bgs. The one-foot soil samples were analyzed for TPH using EPA Method 418.1. No TPH was detected at a detection limit of 20 mg/kg. Based on these results, none of the deeper soil samples were analyzed. Figure 18 shows the locations where the soil samples were collected. Hahn and Associates, the Port's consultant, attributed the presence of petroleum hydrocarbons at the surface to asphalt paving (Hahn and Associates, 1992f). Three ASTs were emptied and removed from the outside of the north wall of Building 4 in 1992.

Prior to 1985, hazardous materials were managed at the point of generation throughout the PSY.

Additional site characterization will be conducted in the WSI Storage Area as part of the RI, because no sampling was conducted after cleanup was completed. The scope of the investigation is discussed later in this section.

Berth 305

Berth 305 was constructed in 1942 along with other berths on the Swan Island Lagoon side of the PSY. The pier at Berth 305 was paved in the mid-1950s and constructed on a combination of wooden pilings and sheet pile wall. Underneath the pier is a catwalk, which extends from Berth 301 to Berth 305. Underneath the catwalk is the Swan Island Lagoon

shoreline that begins approximately 75 to 100 feet west of the eastern edge of the berth.

A series of five sheds are located near the east-end of Berth 305 (see Figure 17). Two of the three westernmost sheds are used as warehouse space by Steelhead Construction. The other westernmost shed is used to store spill containment equipment. The two easternmost sheds are currently used for hazardous waste storage.

During World War II, Berth 305 was used as a new construction finishing pier. After that, Berth 305 was used for outfitting and repair during ship repair work.

In 1994, an environmental assessment was performed for Berths 305 and 306 (Century West, 1994b). The following summarizes the findings of the assessment:

- DEQ LUST file 26-89-0166 states that 190 cubic yards of soil containing petroleum from a 12,700 gallon diesel oil UST No. 2 was removed from an excavation in the south central portion of the berth area (see Figure 17). Three verification samples found no petroleum hydrocarbons were present above detection limits. The DEQ file for the UST was closed on July 24, 1992.
- Between the time that Berth 305 was constructed and the late 1960s, Building 77, the Navy Conversion Building, and Building 35, the Boiler Building, were present at Berth 305.
- Five sheds were located near the east end of Berth 305 (see Figure 17). The following observations were made regarding the three westernmost sheds: bags of sandblast grit were being stored in the north shed, painting of equipment or vehicles appears to have occurred in the center shed, and a work bench lined the wall of the south shed. In their report, Century West stated that a catch basin was located in the center shed. Mr. Alan Sprott of Cascade General recently inspected all five sheds. He was not able to find the catch basin referenced in the Century West report. The only catch basin he observed was located approximately 30 feet to the west of the center shed. The following observations were made regarding the two easternmost sheds: the north shed was used to store full or partially-full 55-gallon drums of oil, secondary containment was provided for drums that were in use, and minor stains were observed on the floor of the "oil shed," also known as the oil storage area; and the south shed contained large spools of cable.
- A mobile, natural gas-fired boiler was located east of the oil shed (see Figure 17).
- There was no significant evidence of surficial staining where empty oil drums were stored south of the oil shed.

A second environmental audit was performed in 1998. No environmental concerns, other than those associated with ship outfitting and repair, were identified during this audit.

No upland investigation is planned in this area, based on the fact that: 1) no environmental concerns were identified in audits conducted in 1994 and 1998, 2) Berth 305 was paved for the last 40 years, and 3) there is no soil underlying most of the berth. No additional sediment investigation will be conducted either, given that sediments near Berth 305 were sampled 1998 by both EPA and jointly by the Port and Cascade General.

Berth 311

Berth 311 is located on the east side of Swan Island Lagoon near the end of the Lagoon. It was constructed in 1969. The berth consists of a concrete pier/lay berth with two access points constructed on wooden pilings. Upland access to Berth 311 is provided by a fenced drive way off of North Basin Avenue. Since it was constructed, Berth 311 was used as a lay-up berth for ships under repair and for hoteling of ships. Because there is no crane service at this berth, only internal work can be conducted on ships. Almost all equipment and materials used during ship repair at Berth 311 remain on the ship.

In July of 1989, one 10,000-gallon unleaded gasoline UST and one 500 gallon used oil UST were decommissioned by permanent removal at the Oregon Freightways facility located north of Berth 311 (Hahn and Associates, 1990). Sampling of the excavated soil confirmed field observations that gasoline and used oil had been released to the surrounding soils. In addition, diesel was found in soil in the vicinity of an AST located to the west of the USTs. Approximately 600 tons or 460 CY of soil were removed from the area formerly occupied by the USTs. Verification soil samples collected beneath the gasoline UST contained between 21 and 66 mg/kg TPH using EPA Method 418.1. The verification soil samples collected beneath the used oil UST contained 280 and 490 mg/kg TPH using EPA Method 418.1. A sample of groundwater that collected in the bottom of the excavation was sampled and found to contain 4 mg/L TPH and no detectable levels of BTEX at a detection limit of 5 ug/L. One year later, approximately 166 cubic yards of soil adjacent to the ASTs was removed. The six verification soil samples collected from the excavation contained between 43 and 250 mg/kg TPH using EPA Method 418.1.

Based on the types of activities conducted at Berth 311, no upland site investigation is planned as part of the RI. Site investigation for sediments will be conducted, however. The scope of the investigation is discussed later in this section.

Sediments

Bulk Chemistry of Surface and Subsurface Sediments

The Portland Shipyard Sediment Investigation Report (Striplin, 1998) provided recommendations for future site investigation of sediments near the PSY. Recommendations were provided the further define bulk sediment chemistry at the surface and at depth. The following surface

sediment chemistry gaps were identified: between Berths 302 and 304, between Berths 306 through 308, and immediately under the dry docks.

Specific subsurface sediment chemistry gaps included: the berths along the PSY side of the Lagoon, on the Willamette River side of the PSY, and adjacent to or in the dry dock basins.

The scope of the site investigation for sediments is presented later in this section.

Bioaccumulation Potential

Table G-2 in Appendix G of the Portland Harbor Sediment Management Plan (PHSMP) includes a list of COIs with respect to bioaccumulation potential. Four of these contaminants were detected in sediments at or near the PSY: TBT, PCBs, mercury, and bis(2-ethylhexyl) phthalate. Of these four contaminants, TBT was detected at the most surface sediment sampling locations and across the broadest range of concentrations (i.e., between 3 and 288 times the average concentration detected in sediment samples collected upstream of the PSY). PCBs were also detected across a broad range of concentrations (i.e., 2 to 132 times the average upstream sediment concentration), but at fewer locations than TBT. Bis(2-ethylhexyl) phthalate was detected at a high percentage of the surface sediment sampling locations, but across a relatively narrow range of concentrations (i.e., 2 to 9 times the average upstream concentration). Finally, mercury was detected at a relatively small number of sampling locations and, with the exception of the far upstream end of the PSY (at Berth 315), it was detected across a narrow range of concentrations (i.e., only 2 to 3 times the average upstream concentration).

Bioaccumulation tests will be performed as part of the RI. The bioaccumulation tests will be run in accordance with the PHSMP. The scope of the tests is discussed later in this section.

Pre-1990 Dredge Spoils

The Site History Section provides an overview of the dredging and dredged material disposal history of the PSY. This same information was provided to DEQ in a letter dated December 9, 1999, including a series of aerial photographs and drawings that illustrate locations that were historically dredged and where certain dredged materials were placed. The information provided to DEQ, and included herein, demonstrates that pre-1990 dredge spoils were either used to fill the shipways when they were abandoned by the Port between 1948 and 1957, or they were placed at the end of the Swan Island Lagoon in accordance with ODSL's 1973 Lower Willamette River Management Plan. The only exception to this was the 1962 in-water placement of dredged materials in Swan Island Lagoon across from Berth 307, in the vicinity of Berth 311.

Sand Blast Grit Composition

Historically, two major types of sandblast grit were used at the PSY: Green Diamond (nickel slag) and Kleen Blast (copper slag). Green

Diamond was the earlier material used at the PSY. After about 1994 or 1995, Kleen Blast was used.

The Material Safety Data Sheet (MSDS) for Green Diamond states that it consists of: 51 percent silica, 33 magnesium oxide, 12 percent iron oxide, 1.5 percent aluminum oxide, 0.5 percent calcium oxide, less than 0.1 percent (i.e., <100 mg/kg) nickel, 0.1 percent (i.e., 100 mg/kg) chromium III oxide, 1.5 percent trace elements and compounds.

The MSDS for Kleen Blast states that it is composed of 38.1 percent silica dioxide, 27.4 percent iron oxide, 22.8 percent calcium oxide, 5.7 percent aluminum oxide, 3.9 percent magnesium oxide, and less than 1 percent fused oxides.

DEQ's File Review Memorandum questions whether the EP Toxicity values presented in Table 4 are representative of past or present used (i.e., spent) abrasive materials. Based on the 1985 and 1998 dates attributed to the test results present in Table 4 of the File Review Memorandum, they must be representative of past, used abrasive materials (likely Green Diamond).

Toxicity Characteristic Leaching Procedure (TCLP) test results provided by Kleen Industrial Services indicate that only barium and cadmium were detected in the extract at 0.81 mg/L and 0.017 mg/L, respectively. Total metals concentrations, as determined by Atomic Absorption Spectroscopy (AAS), were as follows: 1.7 mg/kg for arsenic, 87 mg/kg for barium, 2.6 mg/kg for cadmium, 39 mg/kg for chromium III/IV, 49 mg/kg for cobalt, 1,700 mg/kg for copper, 13 mg/kg for lead, 37 mg/kg for nickel, and 150 mg/kg for zinc. Antimony, beryllium, mercury, molybdenum, selenium, silver, thallium, and vanadium were not detected.

Site Characterization Approach

The SCP is designed so that RI activities will be conducted in two phases. A phased approach will allow for a more focused and efficient site characterization process in that subsequent phases will concentrate only on those areas where additional work activities are deemed necessary. Phase I will focus on determining the nature and extent of contamination at known source areas, identifying the presence of contamination at suspected source areas, characterizing Site hydrogeology, and identifying the presence of contamination in Site groundwater. Phase II will focus on completing the delineation of nature and extent of contamination in soil, groundwater, and sediments, and on collecting additional data required to define hot spots, support ecological and human health risk assessment, and support preparation of a feasibility study. Phase II will also include the identification of sources of contamination to the PSY and parties who contributed to the contamination of the PSY, including current and past operators, contractors and tenants; Kaiser Company, Inc.; the Federal government; potential upstream and downstream sources; and potential sources within the Swan Island Lagoon such as the City of Portland outfalls, Coast Guard facility, U.S. Navy facility and others.

Each phase of the SCP is discussed below.

Phase I Site Investigation

In general, Phase I RI activities will include soil and groundwater sampling in an effort to evaluate known and suspected source areas and to provide needed hydrogeologic information for the Site. Phase I will be further subdivided into two distinct field events. Phase IA will involve the collection of soil samples and screening-level groundwater samples by push probe methodology. Based on the results of the Phase IA investigation, Phase IB will involve the installation of a groundwater monitoring well network at the PSY.

The SCP describes Phase IA RI activities in detail. Phase IB RI activities are described in general, because the specific number and location of monitoring wells to be installed at the PSY will depend upon the Phase IA results. A RI work plan addendum will be submitted to DEQ prior to initiating Phase IB RI activities. All site characterization activities will be conducted in accordance with the Upland Sampling and Analysis Plan (SAP) in Appendix B.

Phase I Site Investigation Objectives

The objectives of the Phase IA site investigation activities are as follows:

- Delineate the nature and extent of soil contamination at known source areas
- Determine if COIs are present in soil in suspected source areas
- Determine the number and location of groundwater monitoring wells to be installed during Phase IB

The objectives of the Phase IB site investigation activities are as follows:

- Characterize Site hydrogeology, including the rate and direction of groundwater flow
- Determine if COIs are present in groundwater beneath known source areas
- Determine the likely future land use for the PSY
- Determine current and likely beneficial water uses
- Determine the locality of the facility
- Identify ecological receptors and sensitive habitats
- Identify preliminary hot spots for soil, groundwater and sediments

Source Areas and COIs

As was discussed earlier, DEQ identified a number of upland data gaps in its File Review Memorandum that could be resolved either through site characterization or through providing the agency with additional information. Each of these data gaps were discussed above, and the following nine upland data gaps will be resolved through site characterization:

- Known Source Areas:
  - Ballast Water Treatment Plant (BWTP), including former BWTP holding ponds now under Building 72
  - N. Channel Avenue Fabrication Site
  - Paint Shed/Blast Booth Area
  - Steam Cleaning Basin (Building 43, 50, and 80 Area)
- Suspected Source Areas
  - Building 73
  - Building 4
  - Electrical Substations
  - Old Boiler (Building 58)
  - Former Hazardous Waste Storage Area (WSI Storage Area)

The COIs for each of these areas were discussed in the Conceptual Site Model Section and are presented in Table 9.

Phase I Upland Site Investigation

The Phase I upland site investigation field program is summarized in Table 25. The analytical testing and quality assurance/quality control program is summarized in Table 26.

In summary, Phase IA will involve the installation of 50 push probe borings in the nine known and suspected source areas to depths ranging from 8 to 40 feet bgs. Screening-level groundwater samples will be collected from 35 of the push probe locations (groundwater is expected to occur at depths of 20 to 30 feet bgs depending on the time of year). Five selected push probe borings will be advanced to 40 feet bgs for hydrogeologic characterization purposes. Selected soil and groundwater samples will be analyzed for COIs appropriate for each source area. Based on the results of the Phase IA investigations, the depths and locations of groundwater monitoring wells will be chosen for installation during Phase IB.

Phase IA Soil Characterization Plan

This section, describing the proposed Phase IA investigation activities relating to soil characterization, is subdivided by source area. The

proposed Phase IA soil sampling program is summarized in Table 27. In general, soil samples will be selected for laboratory analysis at some pre-determined depths, while other samples will be chosen based on field screening indicators and the likelihood of contamination. If field screening does not indicate contamination, then sample selection will be based on other factors such as lithological description (i.e. sandier zones) or vertical location such that samples selected for laboratory analysis will be from within zones of potential preferred contaminant migration.

Ballast Water Treatment Plant

Previous investigations in the vicinity of the BWTP indicate COIs include TPH, PCBs, and metals. These same compounds are also of interest at the former BWTP ponds now located beneath the footprint of Building 72. BTEX and PAHs may also be present in the BWTP area.

Twelve (12) push probes (B-1 through B-12) will be installed in the BWTP (including Building 72) area to characterize subsurface soils. Push probe B-1 will be installed to a depth of 40 feet bgs for hydrogeologic characterization purposes. All other push probes will be installed to depths of approximately 28 to 32 feet bgs, depending on groundwater depth, in an effort to intersect water-saturated soils and sample shallow groundwater. Soil samples will be collected for analysis at depths of 2 feet bgs and at the mean annual groundwater depth, estimated to be at approximately 23 feet bgs, for characterization of the smear zone. The proposed push probe locations at the BWTP area are shown on Figure 19.

Analysis for metals will be conducted for all soil samples in order to characterize the fill material in this area. All soil samples will be analyzed for petroleum hydrocarbons, which will be used as a screening parameter to characterize the extent of contamination, as well as to choose samples for analysis of an expanded list of COIs, these being BTEX, PAHs, and PCBs.

N. Channel Avenue Fabrication Site

The COIs in the N. Channel Avenue Fabrication Site are metals, TPH, PAHs, and VOCs. Previous investigation results indicate that oil-range TPH is present in three locations, near Boring Nos. 3, 4, and 6 completed in 1998 (see Figure 20). It is proposed that the shallow soils in each of these areas be investigated to further characterize the extent of soil contamination.

Four (4) push probes will be installed in each of the three areas: B-13 through B-16 near Boring No. 3, B-17 through B-20 near Boring No. 4 area, and B-21 through B-24 near Boring No. 6. In each area, one push probe (B-13, B-17, and B-21) will be installed to at least 28 feet bgs in the determine the vertical extent of contamination, for the collection of soils containing TPH and to intersect water-saturated soil for the purpose of testing groundwater quality. Soil samples collected from each boring at approximately 2 feet bgs will be selected for analysis. In addition, the 5-foot sample at borings B-13, B-17, and B-21 will be analyzed for the purpose of assessing vertical extent. The remaining four push probe

borings at each area will be installed to minimum depths of 8 feet bgs in order to characterize the lateral extent of contamination.

Three (3) push probes will be installed in one area on the west side of the N. Channel Avenue Fabrication Site where petroleum stained soils were observed during the 1990 ER. The three push probe borings will be installed to minimum depths of 8 feet bgs to determine if COIs are present and to determine the lateral extent of contamination.

The proposed push probe locations in the N. Channel Avenue Fabrication Site are shown on Figure 20.

Field screening indicators will be utilized in an effort to determine the extent of the soil impacts (refer to Appendix B). As such, it is possible that additional push probes, not detailed in this Work Plan, may be installed during Phase IA of the RI to more completely define the extent of contamination. Any additional push probes would be installed in accordance with the upland SAP (Appendix B).

All selected soil samples will be analyzed for petroleum hydrocarbons (TPH-Dx) and metals. A subset of samples will also be analyzed for PAHs and PCBs. Soil samples will not be analyzed for VOCs during this phase of investigation, because methylene chloride was the only VOC detected during previous soil sampling and methylene chloride was only detected in one location (Boring 3) in the near-surface sample. As will be discussed below, VOCs will be sampled in groundwater to determine if releases of VOCs have occurred in this area.

Building 73

Building 73 has always been utilized for surface preparation and has contained a spray booth, paint booth, solvent dip tanks, and a still for solvent reclamation. COIs for Building 73 include TPH, PAHs, VOCs, and metals.

Three (3) push probes (B-28, B-29, and B-30) will be installed in the vicinity of Building 73 to characterize surface soils and provide screening-level groundwater quality data. Boring B-28 will be installed to a depth of 40 feet bgs for hydrogeologic characterization purposes, while borings B-29 and B-30 will be installed to depths of 28 feet bgs or greater in order to collect groundwater samples. The proposed push probe locations in the Building 73 area are shown on Figure 21.

Soil samples collected at 2 feet bgs from each boring will be selected for analysis of TPH, metals and VOCs. Other soil samples will be selected for analysis of PAHs based on field screening results and detected TPH levels.

Building 4

To date, Building 4 has not been the subject of any subsurface investigation activities. Two areas of potential environmental concern were identified at Building 4: 1) the former WSI hazardous material storage area located outside and east of Bays 2 through 5; and 2) abandoned floor drains (now sealed) located inside the building. The Phase IA investigation will focus on the storm sewer line, located to the

west of Building 4, into which these catch basins likely drained. COIs for the Building 4 include: TPH, PAHs, VOCs, and metals.

Five (5) push probes (B-31 through B-35) will be installed in the two targeted areas to characterize surface and subsurface soils, and provide screening-level groundwater quality data. Two push probes (B-31 and B-32) will be placed in the former WSI hazardous material storage area east of Bays 2 through 5. Three push probes (B-33, B-34, and B-35) will be installed along the storm sewer line. The exact location of these push probes will be selected after additional file review and site reconnaissance is completed. Boring B-31 will be installed to a depth of 40 feet bgs for hydrogeologic characterization purposes, while borings B-32 through B-35 will be installed to depths of 28 feet bgs or greater in order to collect groundwater samples. The proposed push probe locations in the Building 4 area are shown on Figure 22.

At a minimum, soil samples will be collected from a depth of 2 feet bgs at B-31 and B-32, and from immediately below the depth of the storm sewer line at B-33, B-34, and B-35. The samples will be analyzed for metals, TPH and VOCs. Other soil samples will be selected for analysis of PAHs based on field screening results and detected TPH levels.

Paint Shed/Blast Booth Area

One paint shed and two blast booths are located to the east of Building 73. COIs in this suspected source area are metals, TPH, PAHs and VOCs.

Five (5) push probes (B-36 through B-40) will be installed to depths of approximately 28 feet bgs in this area to generally characterize surface soils and more importantly provide screening-level groundwater data. At a minimum, soil samples collected at 2 feet bgs will be analyzed for metals and TPH. Other soil samples will be selected for analysis of VOCs and PAHs based on field screening results and detected TPH levels. The proposed push probe locations in this area are shown on Figure 21.

Building 43, 50, and 80 Area (Steam Cleaning Basin)

Previous investigations indicate that oil-range TPH is present in the steam cleaning basin located in the vicinity of Buildings 43, 50, and 80. The proposed investigation in this area is designed to determine the extent of the petroleum impacts, and assess for impacts from PCBs, VOCs, and metals.

Five (5) push probes (B-41 through B-45) will be installed in the steam cleaning basin area to characterize the extent of previously identified soil impacts at Boring No. 14. Boring B-42 will be installed to a depth of 40 feet bgs for hydrogeologic characterization purposes. Boring B-43 will be installed to a depth of 28 feet bgs or greater in order to collect a groundwater sample; and borings B-41, B-44, and B-45 will be installed to a depths of at least 16 feet bgs to characterize the lateral extent of contamination. The proposed push probe locations in the Building 43, 50 and 80 area (Steam Cleaning Basin) are shown on Figure 23.

It is expected that the soil samples from each of the borings at 2 feet bgs will be analyzed for metals, petroleum hydrocarbons (TPH-Dx) and VOCs. In addition, the 5-foot sample at boring B-42 will be analyzed for the purpose of assessing vertical extent. Other soil samples will be selected for analysis of PAHs based on field screening results and detected TPH levels.

Electrical Substations

Eight electrical substations (Substation 1 through Substation 8) are present at the site (Figure 16). Historical information suggests that PCBs may have been present in electrical equipment at the PSY substations, and PCB releases are known to have occurred at Substations 1 and 3. COIs at each of the substations includes TPH as insulating oil (i.e., mineral oil) and PCBs.

Eight (8) near-surface soil samples (approximately 1.0 feet bgs) will be collected at Substation 1 and four (4) near-surface soil samples will be collected at Substations 3 through 9 for a total of 36 soil samples (S-1 through S-36). More samples will be collected in Substation 1 because it is larger than the other substations. No samples will be collected at Substation 2 because it is located inside of Building 60. Because the configuration of each substation varies, soil sample locations will be determined in the field based on areas most likely to contain contamination. Where access permits, push probe equipment will be utilized to collect the soil samples from beneath asphalt cover. Otherwise the samples will be collected with a hand auger at a depth of 1.0 feet bgs. If potential contamination is identified through field screening, the depth of investigation will be increased to determine the vertical extent of contamination. Likewise, additional borings may be added during this phase of investigation to determine the lateral extent of impacts. The soil samples will be analyzed for TPH as insulating oil and PCBs.

Old Boiler (Building 58)

The old boiler was located inside Building 58. It was taken out-of-service since at least 1985. A dry-well was recently discovered by Cascade General near the southwest corner of Building 58. Boiler condensate may have been discharged to this dry well.

One push probe (B-46) will be installed at the location of the dry well to characterize subsurface soils and intersect shallow groundwater. The push probe will be installed to a depth of at least 28 feet bgs. The proposed push probe location is shown on Figure 23.

At a minimum, soil samples will be selected for analysis at depths of 2 feet bgs and at the mean annual groundwater depth, estimated to be at approximately 23 feet bgs, for characterization of the smear zone. The initial soil samples will be analyzed for TPH and metals. Other soil samples may be selected for analysis of PAHs based on field screening results and detected TPH levels.

Former Hazardous Waste Storage Area

As was discussed earlier, hazardous wastes were consolidated and stored in the WSI Storage Area from 1985 through 1994. The Port

conducted a cleanup in this area, but did not collect soil samples. COIs in this area include TPH, PAHs, PCBs, VOCs, and metals.

Four (4) push probes (B-47 through B-50) will be installed to depths of approximately 28 feet bgs in this area to generally characterize subsurface soils and more importantly provide screening-level groundwater data. Soil samples collected at 2 feet bgs will be analyzed for metals, TPH and VOCs. Other soil samples will be selected for analysis of PCBs and PAHs based on field screening results and detected TPH levels. The proposed push probe locations in this area are shown on Figure 21.

Phase I Groundwater Characterization Plan

The primary objectives of the Phase I groundwater investigation are the assessment of Site hydrogeology and groundwater quality in the vicinity of the known and suspected areas. The groundwater characterization plan has two components: 1) screening-level groundwater sampling (Phase IA), and 2) groundwater monitoring well installation activities (Phase IB).

Phase IA Screening-Level Groundwater Sampling

The proposed Phase IA groundwater sampling program is summarized in Table 28. Screening-level groundwater samples will be collected at 35 pre-determined push probe locations from the uppermost groundwater at the site. No vertical profiling of the groundwater is proposed at this time. The selected push probes will be advanced 5 to 10 feet below the water table for groundwater sample collection from a 4-foot screen. Because seasonal water table fluctuations of up to 12 to 15 feet may occur at the Site, the actual depth of sample collection is not known, but is estimated to be between the depths of 28 to 32 feet bgs. The probe locations were selected based on their location relative to known and suspected source areas and proximity to nearby hydrologic features (e.g., Willamette River and Swan Island Lagoon).

All screening-level groundwater samples will be analyzed for VOCs, total metals, and PAHs. Analysis for PCBs in groundwater is not proposed because of the very low solubility of PCBs in water.

Phase 1B Groundwater Monitoring Well Installation

Based on the results of screening-level groundwater sampling, a monitoring well network will be designed and installed at the site for the purpose of monitoring site hydrology and groundwater quality. The actual extent of the monitoring well network cannot be determined until the results of the push probe investigations are received and evaluated. If groundwater is not found to be impacted at any of the known or suspected source areas, then the need for any monitoring wells is arguable. However, conceptually the groundwater monitoring well network will likely consist of approximately 8 shallow-zone groundwater monitoring wells to depths of approximately 35 feet bgs.

Assuming favorable site characterization results, groundwater wells may not be necessary in the vicinity of the N. Channel Avenue Fabrication Site and Building 43, 50, and 80 Area (Steam Cleaning Basin). In this case,

the monitoring network would likely concentrate on the area of the BWTP, Buildings 72 and 73, the Paint Shed/Blast Booth Area, and possibly the Old Boiler (Building 58), WSI Storage Area and Building 4. A conceptual monitoring well network, based on the above conditions, is presented in Figure 24.

Prior to monitoring well network installation, an addendum to the SCP, describing the locations and depths of the proposed monitoring wells, will be submitted to the DEQ for review and approval. The SCP addendum will also include a plan for one round of sampling of the installed wells. Subsequent sampling to provide four quarters of groundwater quality data will be performed during Phase II.

Phase I Storm Water Characterization Plan

Table 29 summarizes the results of the 1998 - 1999 storm water sampling performed by Cascade General in accordance with their NPDES permit No. 1200-Z. Figure 25 shows the three locations where the storm water samples were collected.

The storm water sampling data in Table 29 will be evaluated, along with previous storm water sampling data and analytical results for recently collected storm water samples, during Phase I to determine whether runoff from the Site is a relevant contaminant migration pathway. If this pathway is determined to be relevant, additional storm water sampling may be conducted during Phase II of the RI.

Phase I Air Characterization Plan

No air sampling will be conducted as part of the Phase I RI.

Phase I Surface Water and Sediments

No surface water or sediment sampling will be conducted as part of the Phase I RI.

Phase II Site Investigation

Based on the results of the Phase I site investigation, the Port will complete the additional site investigation activities (i.e., Phase II activities) required to complete the RI. The following provides a description of the additional site investigation activities that can be envisioned at this point in the process. Only a general description of the Phase II upland site characterization activities are provided herein because the scope of these activities is dependant upon the Phase I RI results. More detail is provided on the surface water and sediment sampling activities that will be conducted under Phase II because of the extensive amount of previous sediment investigation work that has already been conducted at the PSY.

Phase II Objectives

The objectives of the Phase II RI are as follows:

- Revise the conceptual model based on the Phase I RI results
- Complete nature and extent delineation for soil, groundwater and sediments
- Complete the identification of hot spots
- Gather data required to complete a baseline human health and ecological risk assessment
- Gather data required to complete a feasibility study
- Identify sources of contamination to the PSY and parties who contributed to the contamination of the PSY

Phase II Upland

Phase II Soil Characterization Plan

Additional soil sampling may need to be conducted during the Phase II RI, depending upon the Phase I results. It is possible that soil sampling will need to be conducted to complete the delineation of the nature and extent of contamination at the known and suspected source areas described above. It is also possible that the Phase IA and IB groundwater sampling results will lead to the identification of other potential source areas that will need to be delineated. Finally, additional soil sampling may be required to complete the identification of hot spots, the baseline human health risk assessment, and the feasibility study.

If additional soil sampling is needed, an addendum to this work plan will be prepared and submitted to DEQ for review and approval prior to initiating the Phase II RI.

Phase II Groundwater Characterization Plan

Phase I of the RI will include the collection of one round of groundwater samples. Phase II of the RI will include the installation and sampling of additional monitoring wells, if needed, to complete the characterization of Site hydrogeology and the delineation of the nature and extent of contamination. Phase II of the RI will also include the collection of three additional quarters of groundwater samples from all monitoring wells installed during Phases I and II. The results of four quarters of groundwater sampling will be used to propose a groundwater monitoring program that may include annual sampling of selected monitoring wells for a specific set of analytes.

Phase II groundwater characterization will also include slug testing of selected monitoring wells for purposes of estimating the hydraulic conductivity of aquifer materials.

If additional groundwater monitoring wells are needed, an addendum to this work plan will be prepared and submitted to DEQ for review and

approval prior to initiating the Phase II RI. If the groundwater monitoring well network installed during Phase I is determined to be sufficient, three additional quarters of groundwater samples will be collected without submitting a work plan addendum.

Phase II Storm Water Characterization Plan

If additional storm water sampling is needed, an addendum to this work plan will be prepared and submitted to DEQ for review and approval prior to initiating the Phase II RI. The upland SAP presented in Appendix B will also need to be revised and provided to DEQ for review and approval.

Phase II Air Characterization Plan

Air sampling may be conducted during the Phase II RI, depending upon the Phase I RI results.

If air sampling is needed, an addendum to this work plan will be prepared and submitted to DEQ for review and approval prior to initiating the Phase II RI. The upland SAP presented in Appendix B will also need to be revised and provided to DEQ for review and approval.

Phase II Surface Water and Sediments

Phase II Surface Water Characterization Plan

No surface water sampling is planned as part of the Phase II RI.

Phase II Sediment Characterization Plan

The following provides a relatively detailed and complete description of the sediment sampling that will be conducted during the Phase II RI. The type, number, and location of sediment samples may, however, be modified depending upon the Phase I RI results. Specifically, if the Phase I RI results indicate that contaminants are migrating to the Willamette River through the groundwater pathway, the sediment characterization plan may be modified to include collection of subsurface sediment samples at likely points of groundwater discharge. Also, if the Phase I RI results indicate that storm water discharges from the Site are a source of contaminants to the sediments, the sediment characterization plan may be modified to include the collection of surface sediment samples near certain storm water outfalls.

Surface grab samples and subsurface cores will be collected to provide the chemical and physical data needed to fill the data gaps discussed earlier. The following outlines the types of samples to be collected, the station location rationale, and the analyses to be performed.

Sampling Design

Surface and subsurface sediment samples will be collected in areas that require additional data to better define lateral and vertical distributions of COIs. The data gaps identified by the DEQ File Review Memorandum and by a thorough review of the data by the Port's consultants identified the six general areas that required further investigation. These areas

include: Swan Island Lagoon between Berths 306 through 308, the PCB area at Berth 302, Berth 311, the dry dock areas, in the small boat basin offshore of the BTWP, and the Willamette River offshore of the BWTP outfall.

The DEQ File Review Memorandum also identified bioaccumulation potential, pre-1990 dredge spoils, and sand blast grit composition as data gaps. The last two were addressed earlier in this section. Bioaccumulation potential will be addressed during Phase II of the RI.

Surface Samples

Eighteen surface sediment stations will be established and sampled during Phase II of the RI (Figure 26). The distribution of these stations among the five areas includes the following:

Swan Island Lagoon between Berth 304 and 308 - Two stations will be placed in this area. One will be located between historical Stations PSY14 and PSY10 and the second will be located between historical Stations PSY10 and PSY3. Both stations are located in areas that could not be sampled during the 1998 PSY Sediment Investigation because vessels were tied up at adjacent berths.

Berth 302 to further define the extent of PCBs near PSY14 - Four stations will be located at and around historic Station PSY14. Surface sediment at Station PSY14 contained the highest concentration of PCBs found during the 1998 PSY Sediment Investigation. Three stations will be placed in close proximity to PSY14, two along side and one offshore, to define the extent of contamination. The site of the original station will be resampled to verify the original PCB concentration detected at PSY14.

Dry Dock Basins - Six surface sediment stations will be placed under Dry Docks 1 through 4. Two will be established under Dry Docks 1, 3 and 4. Sediments under the dry docks have not been sampled since post dredge sampling occurred in 1992 for Dry Docks 1 and 4, and 1994 at Dry Dock 3. Dry Dock 4 and possibly Dry Dock 3 are in depositional areas and the current concentration of COIs in the surface sediment may be different from when the post dredge sampling occurred. A seventh station will be established northwest of the mouth of Dry Dock 4 to further define the extent of contamination beyond historic Station PSY36.

Willamette River side of the Shipyard - Four stations will be established at and around historical Station DM-H. These stations are offshore of the BWTP outfall and contained PAHs and metals.

Berth 311 - One station will be located southeast of Station PSY05 (a surface sediment sample collected during the 1998 PSY Sediment Investigation) to further define the nature and extent of contamination at Berth 311 and near a City of Portland storm water outfall located at the end of the Lagoon.

Subsurface Samples

Nine new subsurface sediment stations will be established and sampled during the RI (Figure 27). The distribution of these stations among the four areas includes the following:

Swan Island Lagoon Adjacent to Berth 311 - One subsurface station will be located southeast of Station PSY05 to define the vertical extent of contamination near Berth 311 and near a City of Portland storm water outfall at the end of the Lagoon.

Berth 302 to further define the extent of PCBs near PSY14 - Two additional subsurface sediment samples will be established at and immediately offshore of historic Station PSY14. A single subsurface sample will be collected at the site of PSY14 to determine the vertical distribution of PCBs at this location. The second subsurface sample will be established immediately offshore of PSY14 to determine the lateral extent of PCBs at depth.

Dry Dock Basins - Three subsurface samples will be established under the dry docks. One sample will be collected under each dry dock to determine vertical distribution of COI in the area. Subsurface sediments under the dry docks have never been sampled and as a result the vertical distribution of COI under the dry docks is unknown. A fourth subsurface station will be established northwest of the mouth of Dry Dock 4 to further define the vertical extent of contamination beyond historic Station PSY36.

Small Boat Basin Offshore of the BWTP - Two subsurface sediment stations will be established between and inshore of these stations to determine the vertical distribution of COIs offshore of the BWTP.

Bioaccumulation Testing

The bioaccumulation potential of PSY sediment will be assessed in accordance with the Portland Harbor Sediment Management Plan (PHSMP). Because DEQ is in the process of developing specific procedures and methods for evaluating the risks posed by bioaccumulative contaminants, the number of sediment samples and COIs to be tested cannot be defined at this time. The proposed approach to bioaccumulation testing will be described in the Phase II RI Work Plan addendum that will be submitted to DEQ for review and approval.

Samples Types

Surface Samples

Eighteen surface samples (plus reference stations) will be collected from in and around the PSY area to close data gaps identified as a result of the 1998 PSY Sediment Investigation (Figure 26). Surface samples will be collected from 0 to 10 cm to represent the biologically active zone of the sediment. These samples will be analyzed to determine their physical and chemical characteristics

Subsurface Samples

Nine subsurface samples will be collected at specific locations in the vicinity of the PSY to close subsurface data gaps (Figure 27). Each core will be visually assessed for obvious sediment layers (e.g., sandblast grit, volcanic ash, changes in sediment grain size). Additional observations, including changes in sediment color, odor, and texture with depth, will be noted on the core log sheets.

Four-foot core sections will be composited and analyzed to assess overall chemical and physical characteristics of historical sediment horizons.

The two cores located in the small boat basin, offshore of the BWTP, will be selected for more detailed core analysis. These cores will be subsampled and chemically and physically characterized at 1-foot intervals as well as at 4-foot intervals. Cores will be taken to the maximum depth possible (i.e., where refusal by the coring device occurs).

Chemical And Physical Analyses

The master list of chemicals of concern for the PSY site will be based on chemicals commonly known to be affiliated with shipyard activities or chemicals that have previously been detected at elevated levels in the vicinity of the PSY. The master list includes: tributyltin (TBT; bulk and porewater), heavy metals, PCBs, volatile and semivolatile organics and pesticides. In order to assess chemical toxicity and bioavailability, samples will also be analyzed for porewater ammonia, total organic carbon (TOC) and total volatile solids (TVS). Grain size will be performed to provide physical data for both bioaccumulation evaluations and possible future remedial feasibility analysis. The master analyte list is provided in Table 30.

Grab Samples

All grab samples will be analyzed for TOC, grain size, total solids, selected total metals, PCBs and semivolatiles. Extra sediment will be collected at each location and archived for future use. All samples will be analyzed for porewater TBT and ammonia.

Core Samples

All core samples will be visually characterized and the results logged. The visual characterization will include presence and depth to distinct sediment layers; sediment type, color, and odor; presence and characterization of sand blast grit; and depth to native sediment if evident.

All subsurface sediment samples will be analyzed for chemical and physical parameters. Four-foot core sections from these locations will be analyzed for selected total metals, semivolatiles, PCBs, TOC and grain size. The three stations that were selected for 1-foot sub-sections will be analyzed for selected total metals, bulk TBT and grain size.

Bioaccumulation Testing

Bioaccumulation testing will be performed on sediment from stations selected based on their concentrations of bioaccumulative COIs. The following two 28-day bioaccumulation tests will be performed: the 28-day oligochaete worm, *Lumbriculus variegates*, test and the 28-day bivalve clam, *Corbicula fluminea,* test.

Current and Reasonably Likely Future and Water Use

Current and reasonably likely future land and water uses in the locality of the facility will be evaluated as part of the RI. Beneficial water uses will be determined by following DEQ's "Guidance for Conducting Beneficial Water Use Determinations at Environmental Cleanup Sites," dated July 1,

1998. Land use will be determined by following DEQ's "Final Guidance, Consideration of Land Use in Environmental Remedial Actions," dated July 1, 1998. Current and reasonably likely future land and water uses will be designated for purposes of identifying hot spots, in accordance with DEQ's "Final, Guidance for Identification of Hot Spots," date April 23, 1998 and for conducting the baseline human health and ecological risk assessments.

RISK ASSESSMENT

This section describes the general scope and approach for the human and ecological risk assessments for the Site. The risk assessments will be conducted in accordance with OAR 340-122-084, DEQ guidance, EPA's "Risk Assessment for Superfund – Human Health Evaluation Manual Part A," and the PHSMP. Because certain procedures and approaches for assessing ecological risks under the PHSMP are currently under development by DEQ, the specific scope and approach for the PSY ecological risk assessment will be described in a risk assessment work plan that will be submitted to DEQ for review and approval prior to initiating the risk assessment.

Human Health Risk Assessment

The proposed human health risk assessment will quantitatively evaluate the complete exposure pathways identified in the conceptual site model. Figure 4 presents a preliminary conceptual site model that includes potential human exposure pathways and receptors. A deterministic human health risk assessment will be performed for both existing and reasonably likely future exposure scenarios.

In accordance with EPA and DEQ guidance, the human health risk assessment will consist of the following four tasks: exposure assessment, toxicity assessment, risk characterization and uncertainty analysis. In the exposure assessment, reasonably likely current and future land uses will be included in the conceptual site model. Exposure point concentrations and exposure factors that reflect site-specific conditions will be estimated for each complete exposure pathway.

Appropriate toxicity criteria will be selected in the toxicity assessment task to quantify carcinogenic and non-carcinogenic risks associated with chemicals of potential concern.

The results of the exposure assessment and toxicity assessment will be combined in the risk characterization task to obtain quantitative estimates of potential cancer and non-cancer risks.

An uncertainty analysis will be performed to determine how different sources of uncertainty affect the risk characterization results.

Exposure Assessment

The objectives of the exposure assessment task are to:

- Develop appropriate exposure units considering the nature, extent, and distribution of contamination and the reasonably likely future land and water uses in the locality of the facility

- Identify contaminants of potential concern for each media of concern
- Develop exposure scenarios based on current and reasonably likely land uses, site features, and potential receptors
- Identify appropriate exposure factors for all complete exposure pathways

The Preliminary Conceptual Site Model section discussed current land use; local geology, hydrogeology and hydrology; and contaminant sources, transport, and fate. Based on the preliminary conceptual model, potential human exposure pathways, exposure routes, and receptors were identified (see Figure 4). This preliminary conceptual site model will be updated throughout each phase of the RI. It will ultimately provide the framework for identifying complete exposure pathways. For an exposure pathway to be complete, there must be an identified source of a chemical or chemicals of potential concern, a release and a transport mechanism from the source, and a receptor or receptors who can come into contact with the contaminants of potential concern (COPCs).

Exposure point concentrations will be developed for each environmental media that a receptor or receptors may contact during the exposure period. Exposure point concentrations will be derived based on Site sampling results or through the use of fate and transport modeling. When basing exposure point concentrations on Site sampling data, one-half the detection limit will be used for analytical results where the chemical concentration is reported as "not detected."

DEQ guidance requires that exposure point concentrations be developed for each COPC in each media at the point of potential contact by a specified receptor. The 90 percent upper confidence limit (UCL) on the arithmetic mean of the environmental concentrations for a COPC will be used to develop an exposure point concentrations. In cases where it can be demonstrated that the environmental concentrations for a particular COPC are not normally distributed, the environmental concentrations will be transformed to log normal values before calculating the 90 percent UCL. If the 90 percent UCL exceeds the maximum concentration detected in an environmental medium, the maximum concentration will be used.

Central tendency estimates (CTE) and reasonable maximum estimates (RME) of exposure will be made using standard, default exposure factors in EPA and DEQ guidance.

Toxicity Assessment

Standard human health risk assessment toxicity databases will be used to derive health-based toxicity criteria. Sources of toxicity criteria will include:

- EPA's Integrated Risk Information System (IRIS)
- EPA's Health Effects Assessment Summary Table (HEAST)
- EPA-NCEA Superfund Health Risk Technical Support Center

- ASTDR minimal risk levels (MRLs)

Toxicity assessments will be performed for two classes of chemicals: carcinogens and non-carcinogens. Reference doses (RfDs) will be used to quantify the toxicity of non-carcinogens. Cancer slope factors will be used to quantify the toxicity of carcinogens.

Risk Characterization

Risk characterization combines the exposure assessment and toxicity assessment results to obtain a quantitative estimate of human health risk. A hazard index (HI) approach will be used to quantify the risk for non-carcinogens. The calculation of a hazard index involves the calculation of a hazard quotient (HQ) for each chemical of potential concern and then summing the chemical-specific HQ's to obtain a HI. The HQ for a particular chemical will be computed as follows:

Hazard Quotient = I/RfD

where:

I = Chemical intake in mg/kg-day

RfD = Reference dose in mg/kg-day

The acceptable risk threshold for a non-carcinogen is HQ or HI less than 1.0.

The excess lifetime cancer risk for carcinogenic chemicals will be computed as follows:

Risk = I x SF

where:

I = Chemical intake in mg/kg-day

SF = Cancer slope factor in $(mg/kg\text{-}day)^{-1}$

The calculated cancer risks for each chemical will be added together for a given exposure pathway to obtain an estimate of the total cancer risk for that exposure pathway. The overall cancer risk will be computed by adding the individual exposure pathway cancer risks.

The acceptable risk thresholds for carcinogens are less than 1×10^{-6} excess lifetime cancer risk for individual carcinogens and less than 1×10^{-5} excess lifetime cancer risk for multiple carcinogens.

Uncertainty Analysis

Uncertainty is inherent in any human health risk assessment. General sources of uncertainty include: the collection and laboratory analysis of environmental samples, exposure factors and scenarios, toxicity criteria, and risk characterization. Each of these sources of uncertainty will be evaluated on a qualitative basis. The general magnitude of the impact of each source of uncertainty will be included in the human health risk assessment, along with a general assessment of whether each source of

uncertainty contributes to an over- or under-estimate of the risk. The uncertainty analysis will put the quantitative risk estimates in context.

Ecological Risk Assessment

The proposed ecological health risk assessment will quantitatively evaluate the complete exposure pathways identified in the conceptual site model. Figure 5 presents a preliminary conceptual site model that includes potential ecological exposure pathways and receptors. A deterministic ecological health risk assessment will be performed for both existing and reasonably likely future exposure scenarios.

The terrestrial portion of the ecological risk assessment will use a tiered approach, starting with the completion of a Level I Scoping Assessment performed in accordance with DEQ's "Guidance for Ecological Risk Assessment," dated April, 1998. A preliminary determination of the locality of the facility with respect to terrestrial receptors and of the presence or absence of terrestrial threatened or endangered species will be made as part of the Level I Scoping Assessment. The results of the Level I assessment will be used to determine the need, if any, for a Level II assessment. Prior to initiating a Level II assessment for terrestrial receptors, the proposed approach to assessing terrestrial ecological risks will be described in a risk assessment work plan that will be submitted to DEQ for review and approval.

The aquatic portion of the ecological risk assessment will be performed in general accordance with the PHSMP. Sediment toxicity to the benthic community will be evaluated based on the bioassays performed during the 1998 Portland Shipyard Sediment Investigation (Striplin, 1998). Risks associated with bioaccumulative contaminants will also be evaluated. Because DEQ is in the process of developing the specific procedures and methods for evaluating the risks posed by bioaccumulative contaminants, the proposed approach to assessing aquatic ecological risks will be described in a risk assessment work plan that will be submitted to DEQ for review and approval.

FEASIBILITY STUDY

The purpose of an FS is to develop and evaluate a range of remedial alternatives for a site. Typically, this range includes a no action alternative that evaluates baseline conditions; an alternative utilizing engineering and institutional controls; a treatment-based alternative; an alternative utilizing excavation and offsite disposal; and one or more alternative(s) utilizing any combination of the preceding alternatives.

In accordance with DEQ's "Final, Guidance for Conducting Feasibility Studies," dated July 1, 1998, for each remedial action, the FS must evaluate:

- The protectiveness of the alternative based upon the standards set forth in OAR 340-122-040
- The feasibility of the alternative based upon balancing of the remedy selection factors which include effectiveness; long-term reliability, implementability, implementation risk, and cost effectiveness
- The extent to which the remedial action alternative treats hot spots of contamination

Any remedial action that is selected or approved by DEQ's Director must be protective, provide a balance of the remedy selection factors, and treat hot spots of contamination to the extent feasible.

A single FS will be prepared for the Site. The FS may address the Site as two operable units: an upland operable unit and a sediment operable unit.

Development of Remedial Action Alternatives

The development of remedial action alternatives will involve the identification of Remedial Action Objectives (RAOs) and general response actions; the identification and screening of remedial action technologies; and the assembly of remedial action alternatives. The development of remedial alternatives will be conducted in accordance with EPA's "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA."

RAOs are medium-specific goals for protecting human health and the environment. The two primary criteria that will be considered when developing RAOs are:

- Remedial actions must achieve the standards for protectiveness specified in OAR 340-122-040(2)
- Remedial actions must treat hot spots of contamination to the extent feasible based on the remedy selection factors

Based on site-specific RAOs, protective preliminary remediation goals (PRGs) and hot spot threshold levels will be calculated.

General response actions are broad categories of actions that will satisfy the RAOs. As was discussed above, the FS will consider a range of general response actions, including: no action, engineering and/or institutional controls, treatment, removal and offsite disposal without treatment, and any combination of the above.

Once the general response actions have been identified, potential remedial technologies will be identified and screened. EPA provides a number of guidance documents and electronic information sources that will be used to identify remedial technologies. The remedial technologies will be screened against the remedy selection balancing factors to identify those technologies that should be eliminated from further consideration.

The final step in the development of remedial alternative process is to assemble the remedial technologies into site-specific remedial alternatives.

Evaluation of Remedial Action Alternatives

Each of the site-specific remedial alternatives will be evaluated against the three requirements listed above.

The protectiveness of each alternative will be based on an assessment of residual risk in accordance with OAR 340-122-084(4). This assessment shall include;

- A quantitative assessment of the risk resulting from concentrations of untreated waste or treatment residuals remaining at the facility
- A qualitative or quantitative assessment of the adequacy and reliability of any institutional or engineering controls
- A demonstration that acceptable risk levels would be attained within the locality of the facility

The preference for treatment of hot spots will be evaluated first by identifying hot spots in accordance with DEQ's "Final, Guidance for the Identification of Hot Spots," dated April 23, 1998. Once hot spots are identified, the feasibility of treating them to the extent feasible will be evaluated based on the five remedy selection factors: effectiveness, long-term reliability, implementability, implementation risk, and reasonableness of cost.

Finally, the balancing of remedy selection factors will be evaluated. This evaluation will be performed on the remedial alternatives both on an individual and comparative basis.

Recommendation of the Remedial Action

The FS will recommend a remedial action alternative from those developed and evaluated in the FS. The following items will be

addressed: periodic reviews, permit exemptions for onsite activities, and designation of points of compliance.

REFERENCES

Caldwell, J.M. and M.C. Doyle. Sediment Oxygen Demand in the Lower Willamette River, Oregon, 1994, USGS, Water-Resources Investigations Report 95-4196, Portland, Oregon, 1995.

Century West Engineering Corporation. Central Utility Building, UST Site Characterization Report, Port of Portland, Portland Ship Repair Yard, prepared for the Port of Portland, January 12, 1994a.

Century West Engineering Corporation. Confidential Report of Findings, Environmental Assessment, Berths 305 and 306, Portland Ship Yard, Swan Island, Portland, Oregon, prepared for the Port of Portland, March 14, 1994b.

Environmental Protection Agency. Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, EPA 540 G-89 004, Office of Emergency and Remedial Response, Washington, DC, October, 1988.

Hahn and Associates. An Environmental Review, Arco Alaska, Inc., Portland Fabrication Site, 5100 N. Channel Avenue, Portland, Oregon, December 8, 1989.

Hahn and Associates. Final Site Visit: Environmental Review, Arco Alaska, Inc. Portland Fabrication Site, Port of Portland, Ship Repair Yard, Portland, Oregon, July 24, 1990a.

Hahn and Associates. Underground Storage Tank Decommissioning, Port of Portland, Oregon Freightways Facility, 5949 North Basin Avenue, Portland, Oregon, prepared for the Port of Portland, August 30, 1990b.

Hahn and Associates. Final Report, Fourth Quarter Groundwater Monitoring Sampling and Analysis, Port of Portland, Ship Repair Yard, Between Buildings #58 and #64, Portland, Oregon, December 6, 1991.

Hahn and Associates. Level II Environmental Assessment, Soils Investigation, Portland Ship Yard, Ballast Water Treatment Plant, Portland, Oregon, prepared for the Port of Portland, February 28, 1992a.

Hahn and Associates. Polychlorinated Biphenyl Inventory and Disposal Recommendations, Port of Portland, Portland Ship Yard, Building #60, Electrical Substation, Portland, Oregon, prepared for the Port of Portland, February 28, 1992b.

Hahn and Associates. Glycol Underground Storage Tank Permanent Decommissioning by Removal, Portland Ship Yard, 5555 N. Channel Avenue, Portland, Oregon, April 9, 1992c.

Hahn and Associates. Letter to Mr. Korvola at the Port of Portland from Mark Wall of Hahn and Associates entitled PCB Sampling Verification Results, Electrical Substation, Portland Ship Yard, Port of Portland, Portland, Oregon, April 9, 1992d.

Hahn and Associates. Phase II Verification Sampling and Analytical Activities, Electrical Substation, Portland Ship Yard, Port of Portland, Portland, Oregon, June 26, 1992e.

Hahn and Associates. Level II Environmental Assessment, Soils Investigation, Portland Ship Yard, 5555 North Channel Avenue, Former Norvac Waste Storage Areas, Portland, Oregon, prepared for the Port of Portland, October 30, 1992f.

Hahn and Associates. Investigation and Excavation of Contaminated Soils, North of Building No. 50, Portland Ship Yard, 5555 N. Channel Avenue, Portland, Oregon, November 9, 1992g.

Hahn and Associates. Soil Investigation, Tank 10 and Pipe Areas, Ballast Water Treatment Plant, Portland Ship Repair Yard, Port of Portland, 5555 Channel Blvd., Portland, Oregon, May 28, 1993.

Hahn and Associates. Soil Remediation, Ballast Water Treatment Plant, Portland Ship Repair Yard, Port of Portland, 5555 N. Channel Avenue, Portland, Oregon, prepared for the Port of Portland, March 31, 1994.

Hahn and Associates. Waste Characterization and Disposal Assistance, Port of Portland, Portland Ship Yard, 5555 N. Channel Avenue, Portland, Oregon, January 16, 1995.

Hart Crowser. Remedial Investigation/Feasibility Study Work Plan, Terminal 4, Slip 3 Upland, Port of Portland, Portland, Oregon, August 18, 1998.

Hartford, S. and W.D. McFarland. Lithology, Thickness, and Extent of Hydrogeologic Units Underlying the East Portland Area, Oregon, USGS, Water-Resources Investigations Report 88-4110, Portland, Oregon, 1989.

Port of Portland. Effects of Waterway Development on Anadromous and Resident Fish in Portland Harbor, Final Report of Research. Edited by D.L. Ward, Oregon Department of Fish and Wildlife for the Port of Portland, Portland, Oregon, 1992.

Striplin Environmental Associates. Portland Shipyard Sediment Investigation Data Report, prepared for the Port of Portland and Cascade General, Inc. by Striplin Environmental Associates, Olympia, Washington, November 9, 1998.

Swanson, R.D. et al. A Description of Hydrogeologic Units in the Portland Basin, Oregon and Washington, USGS, Water-Resources Investigations Report 90-4196, Portland, Oregon, 1993.

TABLE 1
Summary of Soil Sampling Conducted in 1998 in Support of the Proposed PSY Sale

PSY Area	Number of Soil Sampling Locations	Sample Intervals	Analytical Parameters
Channel Avenue Fabrication Site	5	0 – 2 feet 16 – 18 feet or 20 – 22 feet	TPH, PCBs, metals, organic solvent scan
Main Parking Lot	2	0 – 2 feet 16 – 18 feet	TPH, PCBs, metals
BWTP	1	0 – 2 feet 14 – 16 feet	TPH, PCBs, metals
Paint Shed/Blast Booth/Berth 313	5	0 – 2 feet 16 – 18 feet	TPH, PCBs, metals
Substation 3	1	0 – 3 feet 16 – 18 feet	TPH, PCBs, metals
Buildings 43, 50, 80 Area (Steam Cleaning Basin)	1	0 – 2 feet 16 – 18 feet	TPH, PCB, metals
Building 10 and Grit Silo Area	1	0 – 2 feet 16 – 18 feet	TPH, PCBs, metals, organic solvent scan

TABLE 2
Soil Sample Collection Data—Main Parking Lot

Boring No.	Sample Interval	Soil Description	Analytical Parameters
1	0 to 24 inches	0 to 6 inches: Gravel 6 to 24 inches: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	Fine red/brown sand (Groundwater at 17 feet)	TPH, PCB, metals
7	0 to 24 inches	0 to 3 inches: Asphalt 3 to 24 inches: Fine brown sand	TPH, PCB, metals
	16 to 18 feet	Fine brown sand (Groundwater at 17.5 feet)	TPH, PCB, metals

TABLE 3
Analytical Data Results—Main Parking Lot (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
1	0 to 24 inches	Arsenic	0.250	0.500	2.71
		Barium	0.0545	5.00	81.3
		Chromium	0.0470	0.500	12.5
		Lead	0.320	5.00	11.6
	16 to 18 feet	Arsenic	0.250	0.500	1.60
		Barium	0.0545	5.00	84.1
		Chromium	0.0470	0.500	10.5
7	0 to 24 inches	Arsenic	0.250	0.500	2.45
		Barium	0.0545	5.00	158
		Chromium	0.0470	0.500	13.6
		Lead	0.320	5.00	7.00
		Heavy oil range hydrocarbons	13.0†	100†	451†
	16 to 18 feet	Arsenic	0.250	0.500	1.67
		Barium	0.545	5.00	76.7
		Chromium	0.0940	1.00	9.44

*All weights are mg/kg dry unless noted otherwise.
†mg/kg, not reported as "dry"

TABLE 4
Soil Sample Collection Data—Building 10 and Grit Silo Area

Boring No.	Sample Interval	Soil Description	Analytical Parameters
16	0 to 24 inches	0 to 6 inches: Asphalt 6 to 24 inches: Fine red/brown sand	TPH, PCB, metals, solvent scan
	16 to 18 feet	Fine red/brown sand (moist) (Groundwater: none at 18 feet)	TPH, PCB, metals, solvent scan

TABLE 5
Analytical Data Results—Building 10 and Grit Silo Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)	Reporting Limit (mg/kg)	Sample Results (mg/kg)
16	0 to 24 inches	Arsenic	0.250	0.500	2.67
		Barium	0.0545	5.00	120
		Chromium	0.0940	1.00	13.5
	16 to 18 feet	Arsenic	0.250	0.500	1.88
		Barium	0.0545	5.00	87.8
		Chromium	0.0940	1.00	10.2

TABLE 6
Summary of Previous Sediment Investigations Conducted at or Near the PSY

Investigation	Year	No. of Bulk Chemistry Sampling Locations	Analytical Parameters	No. of Biological Sampling Locations
Port Post-Dredge Sampling for Dry Docks 3 and 4	1992 (Dry Dock 4) 1994 (Dry Dock 3)	8 (surface) 6 (surface)	Metals, bulk butyltins, ABNs, pesticides, and PCBs	None
Corps of Engineers Channel Deepening Project	1997	14 (surface)* 11 (cores)*	Metals, ABNs, pesticides, and PCBs	None
EPA/DEQ Portland Harbor Site Assessment	1997	73 (surface)* 21 (cores)*	Metals, ABNs, pesticides, PCBs, bulk organotins, porewater organotins, and titanium	None
Cascade General Independent Sediment Investigation	1997 and 1998	18 (surface) 2 (cores)	Metals, semi-volatile organic compounds, PCBs, and bulk tributyltin	5 amphipod bioassays and a benthic community survey
Port and Cascade General PSY Sediment Investigation	1998	52 (surface) 23 (cores)	Metals, PCBs, volatile organic compounds, semivolatile organic compounds, pesticides, and bulk and porewater tributyltin	52 Micotox, amphipod, and chironomid bioassays

* Number of locations in the vicinity of the PSY

TABLE 7
Historic Maintenance Dredging Activities at the PSY

Year	Dredged Area	Volume of Dredged Material (CY)
1961	Dry Dock 1	4,000[a]
1962	Dry Dock 3 (Berth C)	400[a]
	Berths 306 – 308	3,000[a]
1977	Dry Dock 4	34,000[b]
1981	Dry Dock 3	7,000[a]
1985	Berths 301 - 305	23,667
	Berth 315	153,000[b]
1986	Berths 306-308	1,200
1992	Dry Dock 4	78,000
	Dry Dock 1	17,000
1994	Dry Dock 3	21,000

[a] In-situ volume estimated based on the difference between pre-dredging and post-dredging river bottom elevations.

[b] Construction-related dredging, rather than maintenance dredging.

TABLE 8
Summary of Upland Remedial Actions Conducted at the PSY

PSY Area	Remedial Action	Year	Amount of Soil Removal
Channel Avenue Fabrication Site	Removal of sweepings/sand containing cadmium	1993	60 CY
BWTP	Removal of soil containing TPH and PCBs	1993/1994	90 CY
Central Utility Building	Soil removal around heating oil UST	1998	16 CY[a]
Building 9	UST removal	1989	30 CY
Building 10 (central bay)	UST removal	1989	12 CY
	Removal of PCBs from floor	1992	NA
Building 50	Removal of soil containing oil, diesel and Stoddard solvent	1992	61 CY
Building 58	UST removal	1989	228 CY
Berth 305	UST removal	1989	190 CY

[a] Does not include soil removed in 1994
CY = cubic yards
UD = undocumented
NA = not applicable

TABLE 9
Chemicals of Interest (COIs) for Upland Sources

Source Type	Source	Chemicals of Interest				
		Metals	VOCs	TPH	PAHs	PCBs
Principal						
	Paint Shed/Blast Booth	✓	✓	✓	✓	
	BWTP	✓	✓	✓	✓	✓
	N. Channel Fabrication Site	✓	✓	✓	✓	✓
	Building 43, 50, 80 Area (Steam Cleaning Basin)	✓	✓	✓	✓	
Suspected						
	Building 73	✓	✓	✓	✓	
	Building 4	✓	✓	✓	✓	
	Electrical Substations			✓		✓
	Old Boiler	✓		✓	✓	
	Former Hazardous Waste Storage Area	✓	✓	✓	✓	✓

TABLE 10

Soil Sample Analytical Results – Tank 10 and Pipe Area Samples (Hahn and Associates, 1993)

Date	Sample Number	Depth (in feet bgs)	TPH Concentration (mg/kg)[1]	PCB Concentration (mg/kg)[2]
12/3/92	2223-921202-20	2.0	480	NA
12/3/92	2223-921202-21	3.0	300	NA
3/5/93	2223-930305-26	4.5	<20	<0.1 <0.2
3/5/93	2223-930305-27	5.5	<20	NA
3/17/93	2223-930305-28	5.5	<20	NA
12/3/92	1-2192-92123 (09)	2.0	<20	NA
2/22/93	2223-932202-23 (13)	4.0	98	NA
2/22/93	2223-932202-24 (14)	2.0	53	NA

bgs = below ground surface

[1] EPA Method 418.1

[2] EPA Method 8080, 3350

TABLE 11

Summary of Analytical Results for Soils, Level II Environmental Assessment, Portland Ship Yard, Ballast Water Treatment Plant (Hahn and Associates, 1994)

Date	Sample Number	Depth (in feet bgs)	TPH Concentration (mg/kg)[1]	PCB Concentration (mg/kg)[2]
8/2/93	930802-01	1.5	300	<0.1 - <0.2
8/2/93	930802-02	1.5	220	<0.1 - <0.2
8/2/93	930802-03	4.0	<20	<0.1 - <0.2
8/2/93	930802-04	1.5	<20	<0.1 - <0.2
8/2/93	930802-05	1.5	<20	<0.1 - <0.2
8/2/93	930802-06	4.0	<20	<0.1 - <0.2
8/2/93	930802-07	1.5	73	<0.1 - <0.2
8/2/93	930802-08	1.5	170	<0.1 - <0.2
8/2/93	930802-09	3.0	<20	<0.1 - <0.2
8/2/93	930802-10	1.5	500	<0.1 - <0.2
8/10/93	930810-06	1.5	430	0.24
8/10/93	930810-08	1.5	440	0.4
8/10/93	930810-09	1.5	130	NA
8/10/93	930810-10	4.0	26	NA
8/20/93	930820-01	1.5	240	NA
8/20/93	930820-02	1.5	270	NA
8/20/93	930820-04	1.5	360	NA
9/7/93	930907-02	1.5	100	0.23
9/7/93	930907-03	1.5	ND	NA
9/7/93	930907-04	1.5	ND	NA
9/7/93	930907-05	1.5	81	NA
9/7/93	930907-06	1.5	ND	<0.1 - <0.2
9/7/93	930907-07	1.5	310	NA
9/7/93	930907-10	1.5	300	NA
9/22/93	930922-04	2.0	750	1.4
9/22/93	930922-05	2.0	850	3.1
9/22/93	930922-06	2.5	1,100	2.8
9/22/93	930922-07	2.0	230	<2.5 - <5.0
9/29/93	930929-01	3.5	52	NA

NA = not analyzed
bgs = below ground surface
[1] EPA Method 418.1
[2] EPA Method 8080, 3350

TABLE 12
Soil Sample Collection Data—Tank Farm/Ballast Water Treatment Plant Area

Boring No.	Sample Interval	Soil Description	Analytical Parameters
9	0 to 24 inches	0 to 6 inches: Asphalt 6 to 24 inches: Fine brown sand	TPH, PCB, metals
	14 to 16 feet	Fine brown sand (moist) (Groundwater: none at 16 feet)	TPH, PCB, metals

TABLE 13
Analytical Data Results—Tank Farm/Ballast Water Treatment Plant Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
9	0 to 24 inches	Aroclor 1260	0.00021	0.050	0.0513
		Arsenic	0.250	0.500	1.97
		Barium	0.0545	5.00	89.5
		Chromium	0.0940	1.00	9.88
		Lead	0.320	5.00	9.14
		Heavy oil range hydrocarbons	13.0†	100†	569†
	14 to 16 feet	Aroclor 1260	0.00021	0.050	0.437
		Arsenic	0.250	0.500	4.05
		Barium	0.0545	5.00	144
		Chromium	0.0940	1.00	60.8
		Lead	0.320	5.00	57.0
		Mercury	0.0161	0.100	0.648
		Selenium	0.303	0.500	0.575
		Heavy oil range hydrocarbons	13.0†	100†	1,030†

*All weights are mg/kg dry unless noted otherwise.
†mg/kg, not reported as "dry"

TABLE 14
Soil Sample Collection Data–N. Channel Avenue Fabrication Site

Boring No.	Sample Interval	Soil Description	Analytical Parameters
2	0 to 24 inches	0 to 8 inches: Gravel 8 to 24 inches: Fine red/brown sand	TPH, PCB, metals, organic solvent scan
	20 to 22 feet	Fine red/brown sand (Groundwater 21 feet bgs)	TPH, PCB, metals, organic solvent scan
3	0 to 24 inches	0 to 2 inches: Gravel 2 to 24 inches: Fine red/brown sand	TPH, PCB, metals, organic solvent scan
	16 to 18 feet	16 to 17 feet: Fine gray sand 17 to 18 feet: Gray silty clay (Groundwater: 18 feet)	TPH, PCB, metals, organic solvent scan
4	0 to 24 inches	0 to 4 inches: Gravel 4 to 24 inches: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	16 to 17 feet: Fine brown sand 17 to 17.5 feet: Gray silt 17.5 to 18 feet: Red sand (Groundwater: 17.5 feet)	TPH, PCB, metals
5	0 to 24 inches	0 to 2 inches: Gravel fill 2 to 24 inches: Fine gray sand	TPH, PCB, metals
	16 to 18 feet	Fine gray sand (moist)	TPH, PCB, metals
6	0 to 24 inches	0 to 14 inches: Gravel 14 to 24 inches: Fine brown sand	TPH, PCB, metals
	16 to 18 feet	16 to 17.5 feet: Fine gray sand 17.5 to 18 feet: Gray silt (moist)	TPH, PCB, metals

TABLE 15
Analytical Data Results–N. Channel Avenue Fabrication Site (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
2	0 to 24 inches	Arsenic	0.250	0.500	2.19
		Barium	0.0545	5.00	104
		Chromium	0.0470	0.500	13.3
		Lead	0.320	5.00	12.6
		Heavy oil range hydrocarbons	13.0†	100†	146†
	20 to 22 feet	Arsenic	0.250	0.500	2.73
		Barium	0.0545	5.00	87.6
		Chromium	0.0470	0.500	10.5
3	0 to 24 inches	Methylene chloride	0.019	1.00	1.21
		Arsenic	0.250	0.500	2.44
		Barium	0.0545	5.00	114
		Chromium	0.0470	0.500	12.6
		Lead	0.320	5.00	10.6
		Heavy oil range hydrocarbons	650†	2,500†	3,010†
	16 to 18 feet	Arsenic	0.250	0.500	2.77
		Barium	0.0545	5.00	156
		Chromium	0.0470	0.500	20.9
		Lead	0.320	5.00	11.9
4	0 to 24 inches	Aroclor 1260	0.00021	0.050	0.133
		Arsenic	0.250	0.500	49.8
		Barium	0.0545	5.00	172
		Cadmium	0.0500	0.500	0.935
		Chromium	0.0470	0.500	19.9
		Lead	0.320	5.00	267
		Heavy oil range hydrocarbons	13.0†	100†	2,360†
	16 to 18 feet	Arsenic	0.250	0.500	3.02
		Barium	0.0545	5.00	118
		Chromium	0.0470	0.500	13.7
		Lead	0.320	5.00	5.49
5	0 to 24 inches	Arsenic	0.250	0.500	2.59
		Barium	0.0545	5.00	36.8
		Chromium	0.0470	0.500	6.39
	16 to 18 feet	Arsenic	0.250	0.500	2.14
		Barium	0.0545	5.00	93.5
		Chromium	0.0470	0.500	12.3
6	0 to 24 inches	Arsenic	0.250	0.500	2.41
		Barium	0.0545	5.00	131

TABLE 15
Analytical Data Results–N. Channel Avenue Fabrication Site (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
		Chromium	0.0470	0.500	13.1
		Lead	0.320	5.00	36.9
		Heavy oil range hydrocarbons	13.0†	100†	773†
	16 to 18 feet	Arsenic	0.250	0.500	2.27
		Barium	0.0545	5.00	143
		Chromium	0.235	2.50	16.2
		Lead	0.320	5.00	5.94

*All weights are mg/kg dry unless noted otherwise.
†mg/kg, not reported as "dry"

TABLE 16
Summary of Status of Removed and Active USTs and HOTs at the PSY

Status	UST Identification Number	Location	Installation Date	Size (gallons) and Product	Notes
Removed					
	1	Berth 307 (Crosby & Overton)	5/66	3,000 gasoline	Decommissioned October of 1987; no contamination found
	2 (PSY-18)	Berth 305	–[a]	12,700 diesel	NFA 7/92
	5 (PSY-19)	Southwest of Building 10	–[a]	500 diesel	NFA 7/92
	6	Between Buildings 50 & 43	5/78	500 diesel	NFA 7/92
	7	Between Buildings 50 & 43	5/76	1,000 diesel	NFA 7/92
	8	Between Buildings 50 & 43	5/76	2,000 gasoline	NFA 7/92
	9	East of Building 58	5/61	10,000 fuel oil	Conditional NFA 7/92; groundwater monitoring occurred; site closed
	10 (PSY-17)	East of Building 58	–[a]	250 diesel	Groundwater monitoring occurred; site closed
	11	South of Building 64	5/66	1,000 gasoline	NFA 7/92
	14	North of Building 73	5/78	1,200 glycol	Decommissioned March of 1992; no contamination found
	17	North of Building 4	–[a]	300 diesel	No contamination found
Active					
	12	Central Utility Building	–[a]	20,000 heating oil in concrete vault	Passed leak test in 1998
	13	Central Utility Building	–[a]	20,000 heating oil in concrete vault	Passed leak test in 1998
	15 (PSY-1A)	Card Lock	6/89	6,000 fiberglass gasoline	Double-walled; interstitial leak detection system
	16 (PSY-2A)	Card Lock	6/89	6,000 fiberglass diesel	Double-walled; interstitial leak detection system

[a] Additional Port file review required to determine installation dates

TABLE 17
Summary of Analytical Results for Soil, Subsurface Investigation Between Buildings 58 and 64 (Hahn and Associates, 1991)

Boring/Well Number	Sample Number	Depth (feet bgs)	Date	Analytical Results		
				TPH by Method 418.1 (mg/kg)	Hydrocarbon Identification (HCID)	
					Concentration (mg/kg)	Carbon Range
B-1	B13	15.5	1/9/91	6	NA	NA
B-1	B14	20.5	1/9/91	20	NA	NA
B-2	B23	18.0	1/9/91	<5	NA	NA
B-2	B24	23.0	1/9/91	<5	NA	NA
B-3	B32	15.5	1/10/91	2,800	1,300 1,500	C9-C24 (diesel) C24-C36 (oil)
B-3	B33	18.0	1/10/91	<5	NA	NA
B-3	B34	20.5	1/10/91	<5	NA	NA
B-3	B35	23.0	1/10/91	<5	NA	NA
B-4	B42	13.0	1/10/91	48,000	21,000 27,000	C9-C24 (diesel) C24-C36 (oil)
B-4	B44	18.0	1/10/91	5	NA	NA
B-4	B45	22.5	1/10/91	<5	NA	NA
MW-1	M12	15.5	1/8/91	10	NA	NA
Mw-1	M13	20.5	1/8/91	<5	NA	NA
MW-2	M22	20.5	1/8/91	<5	NA	NA
MW-3	M32	15.5	1/9/91	6	NA	NA
MW-3	M33	20.5	1/9/91	<5	NA	NA

bgs = below ground surface

TABLE 18
Soil Sample Collection Data—Paint Shed and Blast Booth Area

Boring No.	Sample Interval	Soil Description	Analytical Parameters
8	0 to 24 inches	0 to 6 inches: Asphalt 6 to 8 inches: Gravel 8 to 24 inches: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	Fine red/brown sand (Groundwater: 17 feet)	TPH, PCB, metals
10	0 to 24 inches	0 to 6 inches: Asphalt 6 to 17 inches: Gravel 1 to 3 feet: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	16 to 17 feet: Fine red/brown sand 17 to 18 feet: Gray clay (moist and very plastic) (Groundwater: 18 feet)	TPH, PCB, metals
11	0 to 24 inches	0 to 6 inches: Asphalt 6 to 20 inches: Fine red/brown sand 20 to 22 inches: Gray clay 22 to 24 inches: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	Fine red/brown sand (Groundwater at 18 feet)	TPH, PCB, metals
12	0 to 24 inches	0 to 6 inches: Asphalt 6 to 24 inches: Fine brown sand	TPH, PCB, metals, organic solvent scan
	16 to 18 feet	Fine brown sand (moist) (Groundwater: none at 18 feet)	TPH, PCB, metals, organic solvent scan
13	0 to 24 inches	0 to 6 inches: Asphalt 6 to 24 inches: Fine brown sand	TPH, PCB, metals
	16 to 18 feet	Fine red/brown sand (moist) (Groundwater: none at 18 feet)	TPH, PCB, metals

TABLE 19
Analytical Data Results—Paint Shed and Blast Booth Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
8	0 to 24 inches	Arsenic	0.250	0.500	2.13
		Barium	0.0545	5.00	88.3
		Chromium	0.0940	1.00	11.9
	16 to 18 feet	Arsenic	0.250	0.500	2.52
		Barium	0.0545	5.00	94.2
		Chromium	0.0940	1.00	12.4
10	0 to 24 inches	Arsenic	0.250	0.500	3.46
		Barium	0.0545	5.00	254
		Chromium	0.0940	1.00	20.9
		Lead	0.320	5.00	7.14
		Heavy oil range hydrocarbons	13.0†	100†	644†
		Selenium	0.303	0.500	0.775
	16 to 18 feet	Arsenic	0.250	0.500	3.60
		Barium	0.0545	5.00	217
		Chromium	0.0940	1.00	25.3
		Lead	0.320	5.00	9.67
11	0 to 24 inches	Arsenic	0.250	0.500	3.65
		Barium	0.0545	5.00	187
		Chromium	0.0940	1.00	25.1
		Lead	0.320	5.00	8.99
		Heavy oil range hydrocarbons	13.0†	100†	365†
		Selenium	0.303	0.500	0.715
	16 to 18 feet	Arsenic	0.250	0.500	2.48
		Barium	0.0545	5.00	163
		Chromium	0.0940	1.00	15.9
12	0 to 24 inches	Arsenic	0.250	0.500	2.92
		Barium	0.0545	5.00	130
		Chromium	0.0940	1.00	15.9
		Lead	0.320	5.00	5.66
		Heavy oil range hydrocarbons	13.0†	100†	180†
	16 to 18 feet	Methylene chloride	0.019	1.00	1.05
		Arsenic	0.250	0.500	2.19
		Barium	0.0545	5.00	109
		Chromium	0.0940	1.00	13.9
13	0 to 24 inches	Arsenic	0.250	0.500	3.67
		Barium	0.0545	0.500	152
		Chromium	0.0940	1.00	14.8
		Lead	0.320	5.00	6.47
		Heavy oil range hydrocarbons	13.0†	100†	198†
	16 to 18 feet	Arsenic	0.250	0.500	2.14

TABLE 19
Analytical Data Results—Paint Shed and Blast Booth Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
		Barium	0.0545	5.00	88.8
		Chromium	0.0940	1.00	10.6

*All weights are mg/kg dry unless noted otherwise.
†mg/kg, not reported as "dry"

TABLE 20
Soil Sample Collection Data—Buildings 43, 50, 80 Area

Boring No.	Sample Interval	Soil Description	Analytical Parameters
14	0 to 24 inches	0 to 6 inches: Asphalt 6 to 24 inches: Fine red/brown sand	TPH, PCB, metals
	16 to 18 feet	Fine red/brown sand (moist) (Groundwater: none at 18 feet)	TPH, PCB, metals

TABLE 21
Analytical Data Results—Buildings 43, 50, 80 Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)*	Reporting Limit (mg/kg)*	Sample Results (mg/kg)*
14	0 to 24 inches	Arsenic	0.250	0.500	2.85
		Barium	0.0545	5.00	117
		Chromium	0.0940	1.00	13.7
		Lead	0.320	5.00	14.6
		Heavy oil range hydrocarbons	13.0†	100†	558†
	16 to 18 feet	Arsenic	0.250	0.500	2.02
		Barium	0.0545	5.00	86.0
		Chromium	0.0940	1.00	11.7

*All weights are mg/kg dry unless noted otherwise.
†mg/kg, not reported as "dry"

TABLE 22
Location, Construction Date, Current PCB Content, and Condition of PSY Substations

Substation	Location	Construction Date	Number and Type of Existing Transformers	PCB Content of Insulating Fluid in Existing Transformers	Site Condition
1	South of Building 60	~1950	3 transformers: 1500 KVa, 1000 KVa & 1000 KVa RTEMP	No PCBs as of 1992	Asphalt paved and fenced
2	Inside northwest corner of Building 60	~1952 (same time as Building 60)	None	No PCBs as of 1992	Inside building
3	Head of Dry Dock 1	Between 1961 and 1967	2 transformers: 1500 KVa & 1000 KVa silicon (non-PCB)	No PCBs as of 1992	Asphalt paved and fenced; steel-beam structure with corrugated metal siding and roof
4	East of Building 43		1 transformer: 1000 KVa silicon (non-PCB)	No PCBs as of 1985	Steel-beam structure with corrugated metal siding and roof; paved floor
5	Berth 304 west of the oxygen storage area	1989	2 transformers: 1500 KVa & 1000 KVa silicon (non-PCB)	Retrofitted with non-PCB equipment in early 1990s	Asphalt-paved with transformers and breakers housed in metal cabinet
6	Berth 305 west of Building 6	1988	2 transformers: 1500 KVa & 1000 KVa RTEMP (non-PCB)	Retrofitted with non-PCB equipment in early 1990s	Asphalt-paved with transformers and breakers housed in metal cabinet
7	Between Berths 312 and 313	~1979 (same as berth construction)	1 transformer: 750 KVa oil (no PCB data)	No PCBs as of 1992	Paved
8	Between Berths 313 and 314	~1979 (same as berth construction)	1 transformer: 2000 KVa RTEMP (non-PCB)	No PCBs as of 1992	Paved

TABLE 23
Soil Sample Collection Data—Substation 3 Area

Boring No.	Sample Interval	Soil Description	Analytical Parameters
15	0 to 36 inches	0 to 8 inches: Asphalt 8 to 12 inches: Gravel 12 to 24 inches: Fine red sand 24 to 36 inches: Fine gray sand	TPH, PCB, metals
	16 to 18 feet	Coarse brown sand (moist) (Groundwater: none at 18 feet)	TPH, PCB, metals

TABLE 24
Analytical Data Results—Substation 3 Area (Detected Constituents Only)

Boring No.	Sample Interval	Detected Analyte	Detection Limit (mg/kg)	Reporting Limit (mg/kg)	Sample Results (mg/kg)
15	0 to 36 inches	Arsenic	0.250	0.500	1.57
		Barium	0.0545	5.00	55.6
		Chromium	0.0940	1.00	8.10
		Lead	0.320	5.00	18.1
	16 to 18 feet	Arsenic	0.250	0.500	1.19
		Barium	0.0545	5.00	77.3
		Chromium	0.0940	1.00	7.51

TABLE 25
Phase I RI Field Program Summary

Area of Investigation	Number of Push Probe Borings with Soil Samples	Initial Number of Near-Surface Soil Samples [1] (0-2 feet bgs)	Initial Number of Subsurface Soil Samples [1]	Number of Screening-Level Groundwater Sample Locations	Tentative Number of Groundwater Monitoring Wells
Ballast Water Treatment Plant (BWTP)	12	12	12	12	4
N. Channel Fabrication Site	15	15	3	3	-
Building 73 (surface preparation and painting area)	3	3	-	3	1
Building 4	5	2	3	5	-
Paint Shed/Blast Booth Area	5	5	-	5	1
Building 43, 50, 80 Area (Steam Cleaning Basin)	5	5	1	2	-
Electrical Substations	-	36	-	-	-
Old Boiler Area	1	1	1	1	1
Former Hazardous Waste Storage Area	4	4	-	4	1
Totals	50	83	20	35	8

bgs = below ground surface

[1] Additional samples may be selected for analysis based on field screening indicators or initial analytical results

TABLE 26
Proposed Phase IA Laboratory Analysis Summary Continued

Area of Investigation	Investigative Method	Number Soil Borings	Sample Matrix	Analytical Parameters	Tentative Number of Soil Samples to be Analyzed				
					Investigative Samples	Field QA/QC Samples			Matrix Total
						Duplicate	Trip Blank	Equip. Blank	
Building 43, 50, 80 Area (Steam Cleaning Basin)	Push Probe	5	Soil	Metals	6	1			7
				TPH-Dx	6	1			7
				PAHs	3	1			4
				VOCs	6	1			7
		2	Groundwater	Metals	2				2
				PAHs	2				2
				VOCs	2				2
Electrical Substations	Hand Auger	36	Soil	TPH	36	2			38
				PCBs	36	2			38
Old Boiler Area	Push Probe	1	Soil	Metals	2				2
				TPH	2				2
				PAHs	1				1
		1	Groundwater	Metals	1				1
				PAHs	1				1
				VOCs	1				1
Former Hazardous Waste Storage Area	Push Probe	4	Soil	Metals	4	1			5
				TPH	4	1			5
				PAHs	2	1			3
				PCBs	2	1			3
				VOCs	4	1			5
		4	Groundwater	Metals	4	1		1	6
				PAHs	4	1		1	6
				VOCs	4	1	1	1	7

bgs = below ground surface
BTEX = benzene, toluene, ethylbenzene, and xylene
PAHs = polynuclear aromatic hydrocarbons
PCBs = polychlorinated biphenyls

QA/QC = quality assurance / quality control
TPH = total petroleum hydrocarbons by hydrocarbon identification (HCID) method
TPH-Dx = diesel and oil-range total petroleum hydrocarbons
VOCs = volatile organic compounds

TABLE 26
Proposed Phase IA Laboratory Analysis Summary

Area of Investigation	Investigative Method	Number Soil Borings	Sample Matrix	Analytical Parameters	Tentative Number of Soil Samples to be Analyzed				
					Investigative Samples	Field QA/QC Samples			Matrix Total
						Duplicate	Trip Blank	Equip. Blank	
Ballast Water Treatment Plant (BWTP)	Push Probe	12	Soil	Metals	24	2			26
				TPH	24	2			26
				BTEX	12	1			13
				PAHs	12	1			13
				PCBs	12	1			13
		12	Groundwater	Metals	12	1		1	14
				BTEX	12	1	1	1	15
				PAHs	12	1		1	14
N. Channel Fabrication Site	Push Probe	15	Soil	Metals	18	2			20
				TPH-Dx	18	2			20
				PAHs	9	1			10
				PCBs	9	1			10
		3	Groundwater	Metals	3	1		1	5
				PAHs	3	1		1	5
				VOCs	3	1	1	1	6
Building 73	Push Probe	3	Soil	Metals	3				3
				TPH	3				3
				PAHs	2				2
				VOCs	3				3
		3	Groundwater	Metals	3				3
				PAHs	3				3
				VOCs	3				3
Building 4	Push Probe	5	Soil	Metals	5				5
				TPH	5				5
				PAHs	2				2
				VOCs	5				5
		5	Groundwater	Metals	5				5
				PAHs	5				5
				VOCs	5				5
Paint Shed/Blast Booth Area	Push Probe	5	Soil	Metals	5	1			6
				TPH	5	1			6
				PAHs	2	1			3
				VOCs	2	1			3
		5	Groundwater	Metals	5	1		1	7
				PAHs	5	1		1	7
				VOCs	5	1	1	1	8

bgs = below ground surface
BTEX = benzene, toluene, ethylbenzene, and xylene
PAHs = polynuclear aromatic hydrocarbons
PCBs = polychlorinated biphenyls

QA/QC = quality assurance / quality control
TPH = total petroleum hydrocarbons by hydrocarbon identification (HCID) method
TPH-Dx = diesel and oil-range total petroleum hydrocarbons
VOCs = volatile organic compounds

TABLE 27
Proposed Phase IA Soil Sampling Program

Area	Push Probe Boring Numbers	Proposed Boring Depths (feet bgs)	Collect Groundwater Sample	Tentative Number of Soil Samples to be Analyzed	Soil Sample Method	Tentative Soil Samples Selected for Analysis (feet bgs)	Analytical Parameters
Ballast Water Treatment Plant (BWTP)	B-1	40 [1]	Yes	2	Continuous Soil Core	2' & 23' bgs (mean water table); Others based on field screening	Metals, TPH Expanded List [3]: BTEX, PAHs, PCBs
	B-2 to B-12	28 - 32 [2]	Yes	22			
N. Channel Fabrication Site	B-13	40 [1]	Yes	2	Continuous Soil Core	2 ' bgs; 5' bgs @ B-13, B-18, B-23; Others based on field screening and analytical results	Metals, TPH-Dx Expanded Parameters [3]: PAHs, PCBs
	B-14 to B-16	8	No	3			
	B-17	28 - 32 [2]	Yes	2			
	B-18 to B-20	8	No	3			
	B-21	28 - 32 [2]	Yes	2			
	B-22 to B-24	8	No	3			
	B-25 to B-27	8	No	3			
Building 73	B-28	40 [1]	Yes	1	Continuous Soil Core	2 ' bgs; Others based on field screening and analytical results	Metals, TPH, VOCs Expanded List [3]: PAHs
	B-29, B-30	28 - 32 [2]	Yes	2			
Building 4	B-31	40 [1]	Yes	1	Continuous Soil Core	2 ' bgs or below utility trench depth; Others based on field screening and analytical results	Metals, TPH, VOCs Expanded List [3]: PAHs
	B-32 to B-35	28 - 32 [2]	Yes	4			
Paint Shed/Blast Booth Area	B-36 to B-40	28 - 32 [2]	Yes	5	Continuous Soil Core	2 ' bgs; Others based on field screening and analytical results	Metals, TPH Expanded List [3]: PAHs, VOCs
Building 43, 50, 80 Area (Steam Cleaning Basin)	B-42	40 [1]	Yes	2	Continuous Soil Core	2 ' bgs; 5' bgs @ B-41; Others based on field screening and analytical results	Metals, TPH-Dx, VOCs Expanded List [3]: PAHs
	B-43 to B-45	28 - 32 [2]	Yes	1			
	B-41, B-44 and B-45	16	No	3			
Electrical Substations	S-1 to S-36	2	No	36	Hand Auger or Push Probe	1 ' bgs; Others based on field screening and analytical results	TPH, PCBs
Old Boiler Area	B-46	28 - 32 [2]	Yes	2	Continuous Soil Core	2' & 23' bgs (mean water table); Others based on field screening	Metals, TPH Expanded List [3]: PAHs
Former Hazardous Waste Storage Area	B-47 to B-50	28 - 32 [2]	Yes	4	Continuous Soil Core	2 ' bgs; Others based on field screening and analytical results	Metals, TPH, VOCs Expanded List [3]: PAHs, PCBs

bgs = below ground surface
BTEX = benzene, toluene, ethylbenzene, and xylene
PAHs = polynuclear aromatic hydrocarbons
PCBs = polychlorinated biphenyls
TPH = total petroleum hydrocarbons by HCID method and follow up quantitation, unless otherwise indicated
TPH-Dx = TPH method for diesel- and oil-range petroleum hydrocarbons
VOCs = volatile organic compounds

[1] = For hydrogeologic characterization
[2] = Borings will be installed to a minimum of 28 feet bgs, but will be advanced deeper if necessary to collect a groundwater sample
[3] = Approximately 1/2 of the samples will be analyzed for expanded parameters; samples will be selected based on the highest detected TPH concentrations

TABLE 28
Proposed Phase IA Groundwater Sampling Program

Area	Push Probe Boring Numbers	Proposed Boring Depths (feet bgs)	Estimated Screen Interval (feet bgs)	Tentative Number of Groundwater Samples to be Analyzed	Analytical Parameters
Ballast Water Treatment Plant (BWTP)	B-1	40 [1]	24 - 28	1	Metals, PAHs, VOCs
	B-2 to B-12	28 - 32 [2]	24 - 28	11	
North Channel Fabrication Site	B-13	40 [1]	24 - 28	1	Metals, PAHs, VOCs
	B-18, B-23	28 - 32 [2]	24 - 28	2	
Building 73	B-28	40 [1]	24 - 28	1	Metals, PAHs, VOCs
	B-29, B-30	28 - 32 [2]	24 - 28	2	
Building 4	B-31	40 [1]	24 - 28	1	Metals, PAHs, VOCs
	B-32 to B-35	28 - 32 [2]	24 - 28	4	
Paint Shed/Blast Booth Area	B-36 to B-40	28 - 32 [2]	24 - 28	5	Metals, PAHs, VOCs
Building 43, 50, 80 Area (Steam Cleaning Basin)	B-41	40 [1]	24 - 28	1	Metals, PAHs, VOCs
	B-42	28 - 32 [2]	24 - 28	1	
Old Boiler Area	B-46	28 - 32 [2]	24 - 28	1	Metals, PAHs, VOCs
Former Hazardous Waste Storage Area	B-47 to B-50	28 - 32 [2]	24 - 28	4	Metals, PAHs, VOCs

bgs = below ground surface PAHs = polynuclear aromatic hydrocarbons VOCs = volatile organic compounds

[1] = For hydrogeologic characterization

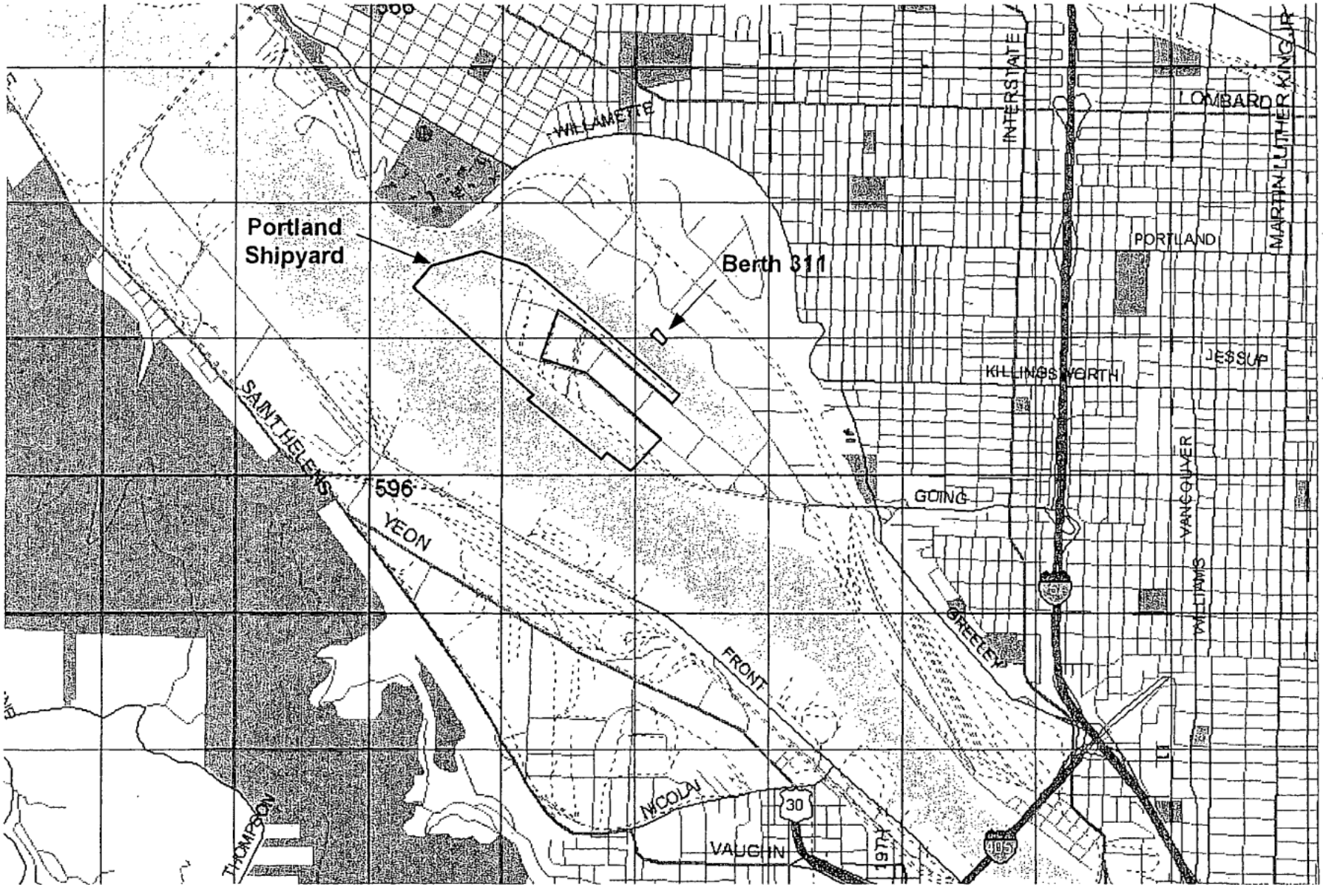
[2] = Borings will be installed to a minimum of 28 feet bgs, but will be advanced deeper if necessary to collect a groundwater sample

TABLE 30
Target Analytes and Quantitation Limits (QLs) for Sediment Samples

Analytes	Required Quantitation Limit
Metals Total (mg/kg dry wt.)	
(EPA 3050, 6010, 7000)	
Antimony	20
Arsenic	57
Cadmium	0.96
Chromium	270
Copper	81
Lead	66
Mercury	0.21
Nickel	140
Silver	1.2
Zinc	160
ButylTins as Ion (ug/l)	
(GC/MS)	
Tributyltin (ug/l interstitial water)	0.05
(GC FPD)	
Bulk Butyltin (ug/kg dry weight)	1
Conventional Analytes	
Total Solids (%)	Std.CAS
Ammonia in Interstitial Water (mg/l)	Std.CAS
Grain Size - ASTM-D-442-63/PSEP	Std.CAS
Volatile Solids (% Not Analyzed)	Std.CAS
TOC (% Dry Weight)	Std.CAS

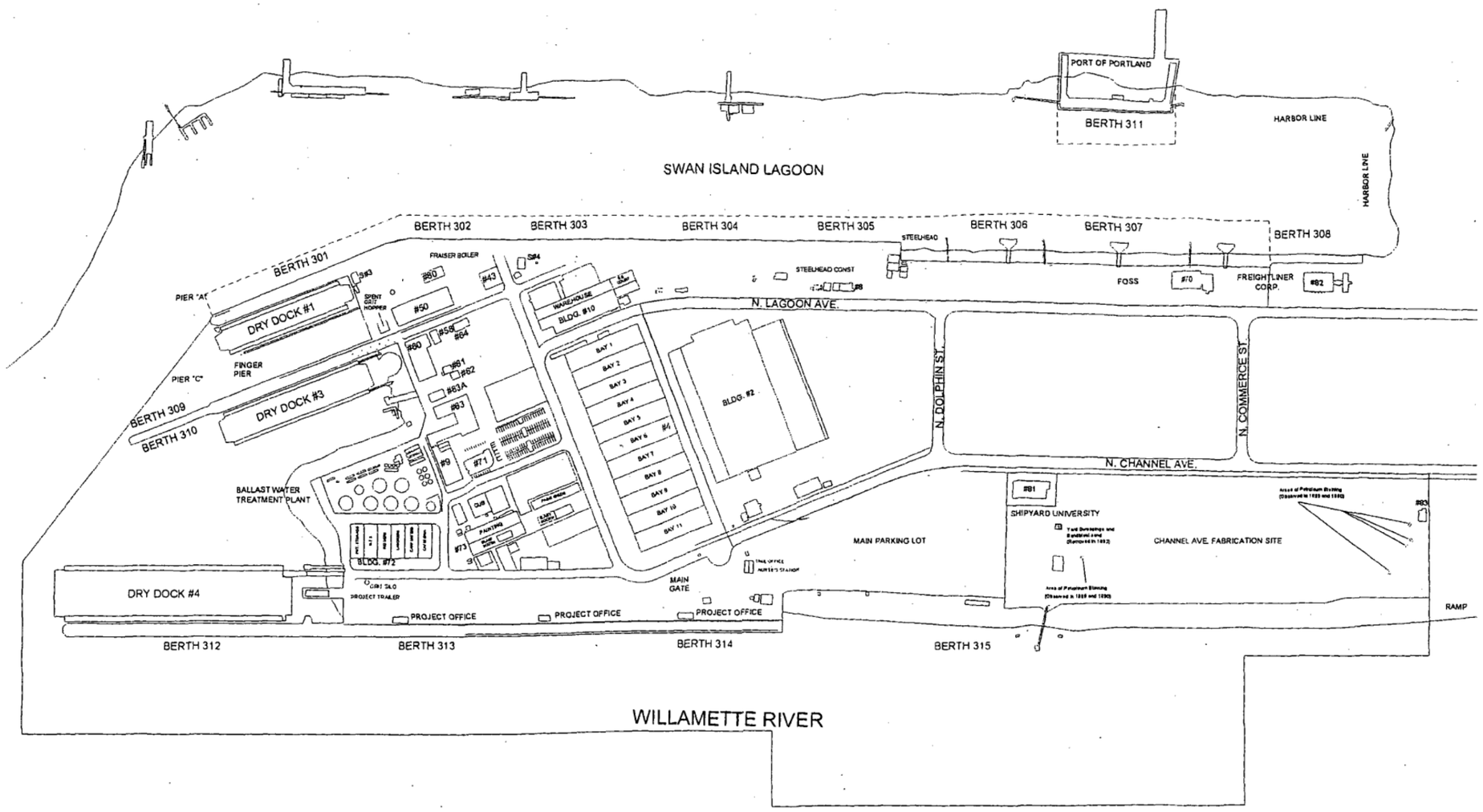
TABLE 30
Target Analytes and Quantitation Limits (QLs) for Sediment Samples

Analytes	Required Quantitation Limit
Pesticides (ug/kg):	
(EPA 8081)	
Aldrin	10
alpha-BHC	10
beta-BHC	10
delta-BHC	10
gamma-BHC (Lindane)	10
Chlordane	10
4,4'-DDD	6.9
4,4'DDE	6.9
4,4'-DDT	6.9
Dieldrin	10
Endosulfan	10
Endosulfan II	10
Endosulfan Sulfate	10
Endrin	10
Endrin Aldehyde	10
Endrin Ketone	10
Heptachlor	10
Heptachlor Epoxide	10
Methoxychlor	10
Toxaphene	20
PCBs (ug/kg)	
(EPA 8082)	
Aroclor 1016	33
Aroclor1221	66
Aroclor1232	33
Aroclor1242	33
Aroclor1248	33
Aroclor1254	33
Aroclor1260	33
Total PCB	



Portland,
Oregon

Figure 1
Location Map
Portland Shipyard
BRIDGEWATER GROUP, INC.



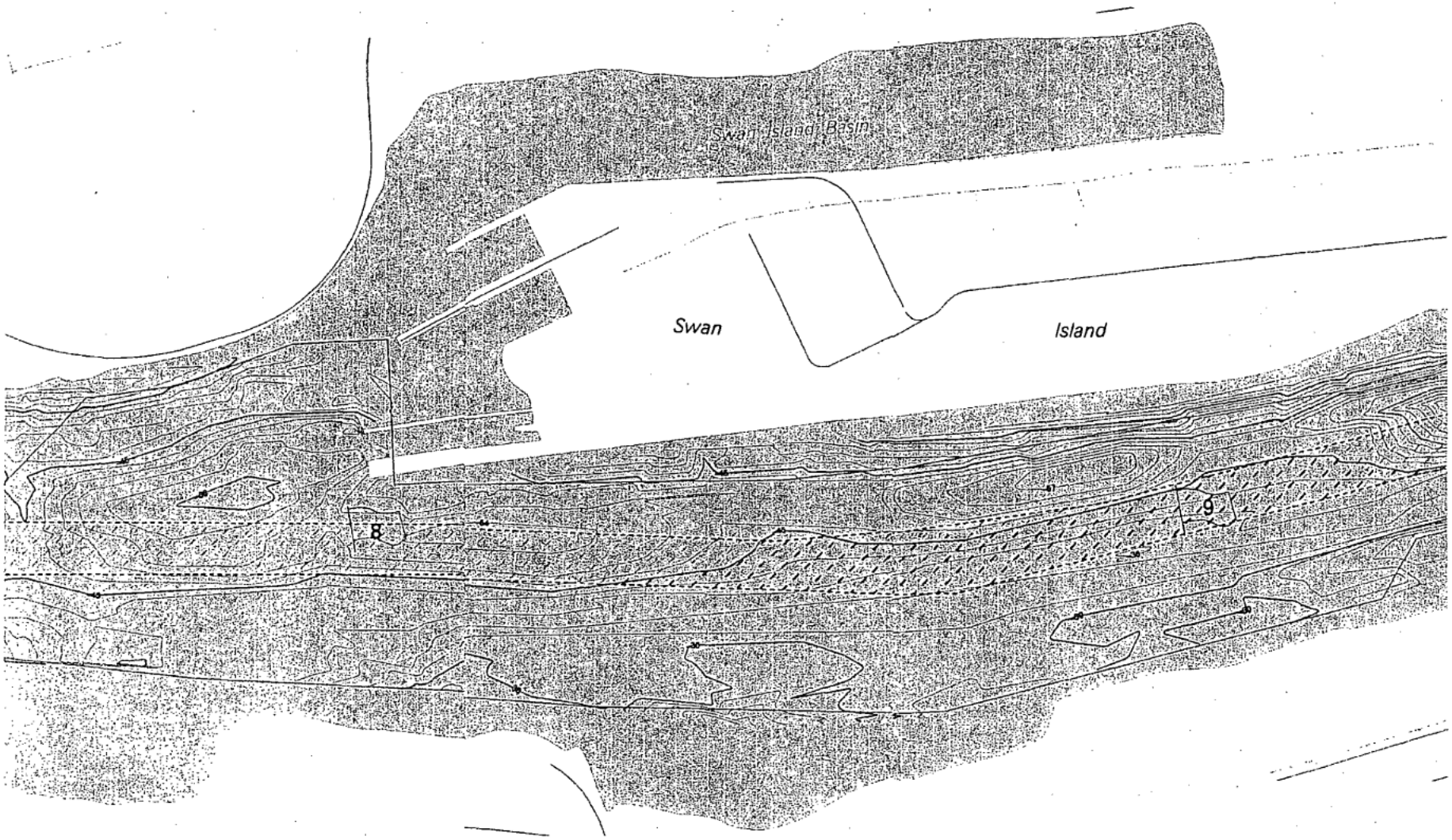
Legend:

——— Approximate Site Boundary

— – Submerged Lands Leased from ODSL

0 ft. 250 ft. 500 ft. 1000 ft.

Figure 2
Site Map
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Source: COE 1998 Channel Deepening Project Bathymetric Maps

Figure 3
Willamette River Bathymetry
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

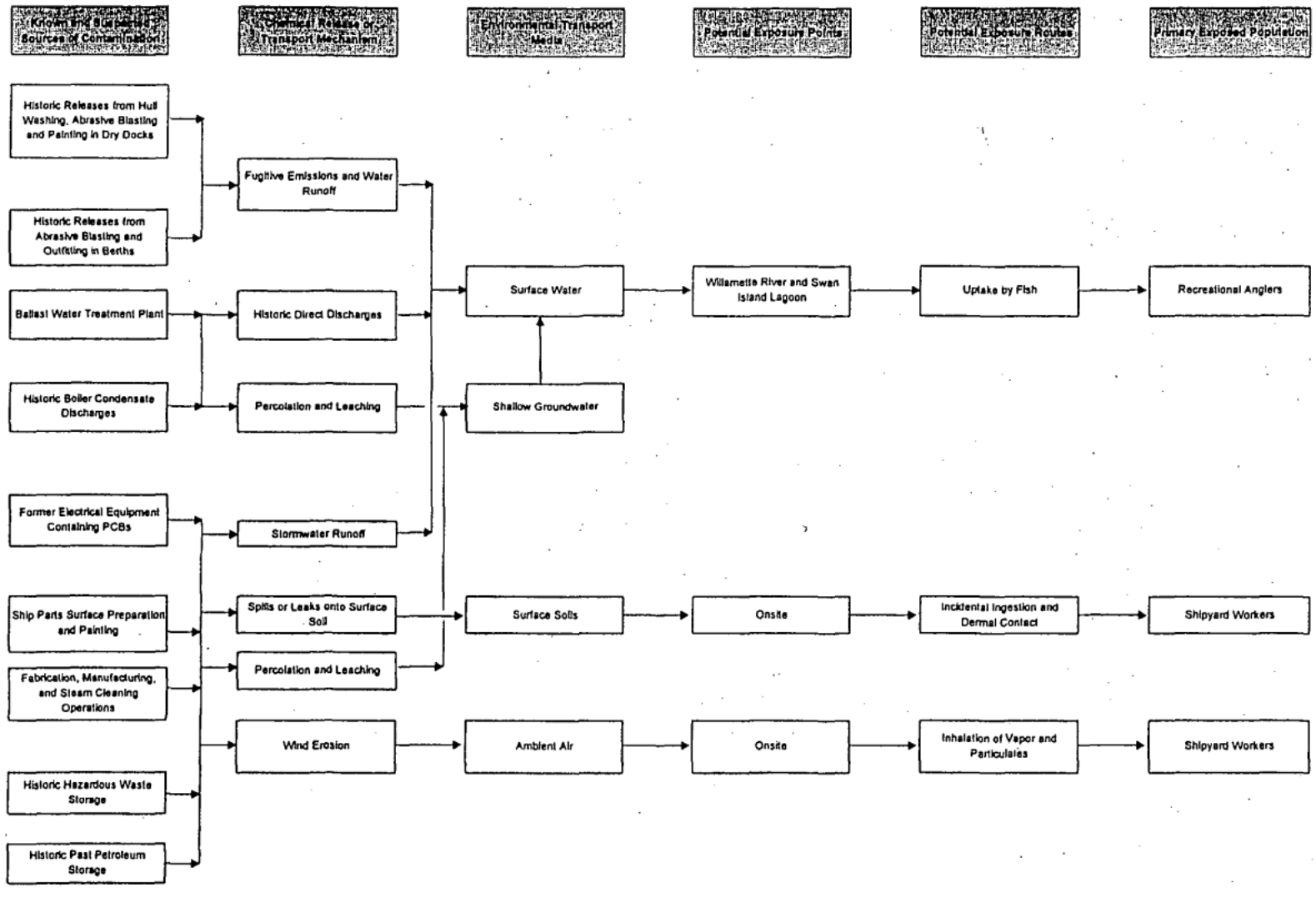


Figure 4
Conceptual Site Model for Potential Human Receptors
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

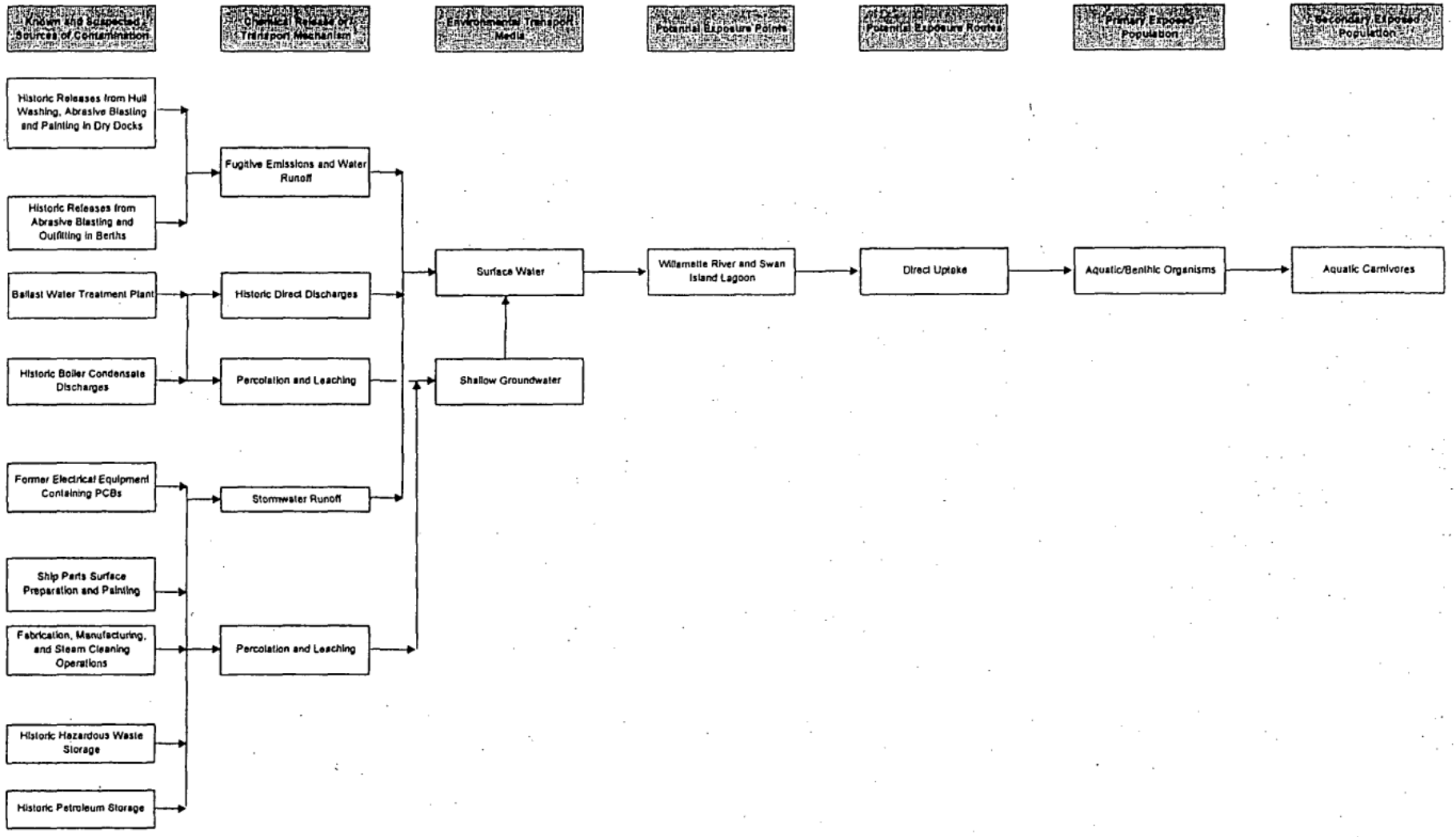


Figure 5
Conceptual Site Model for Potential Ecological Receptors
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

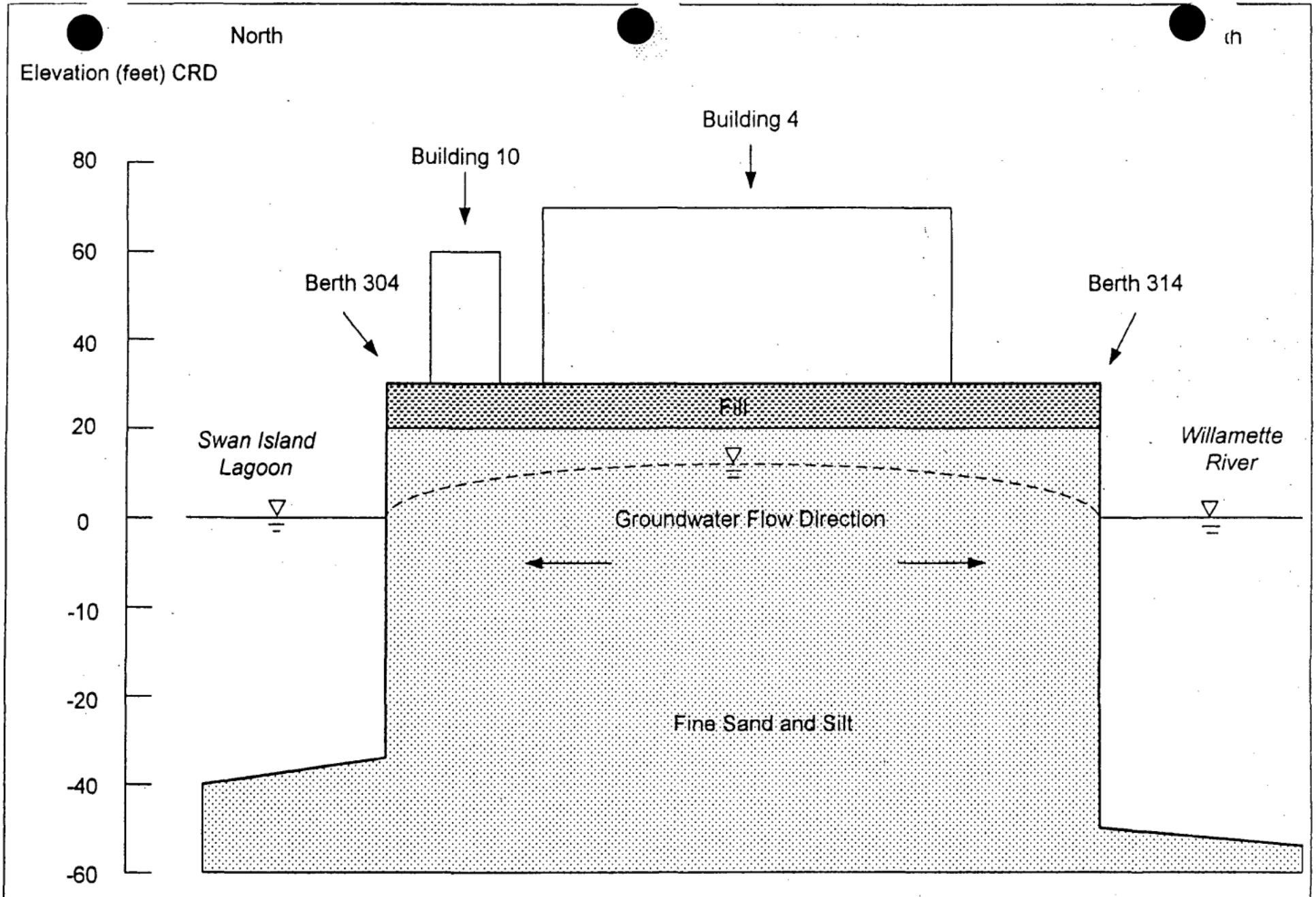
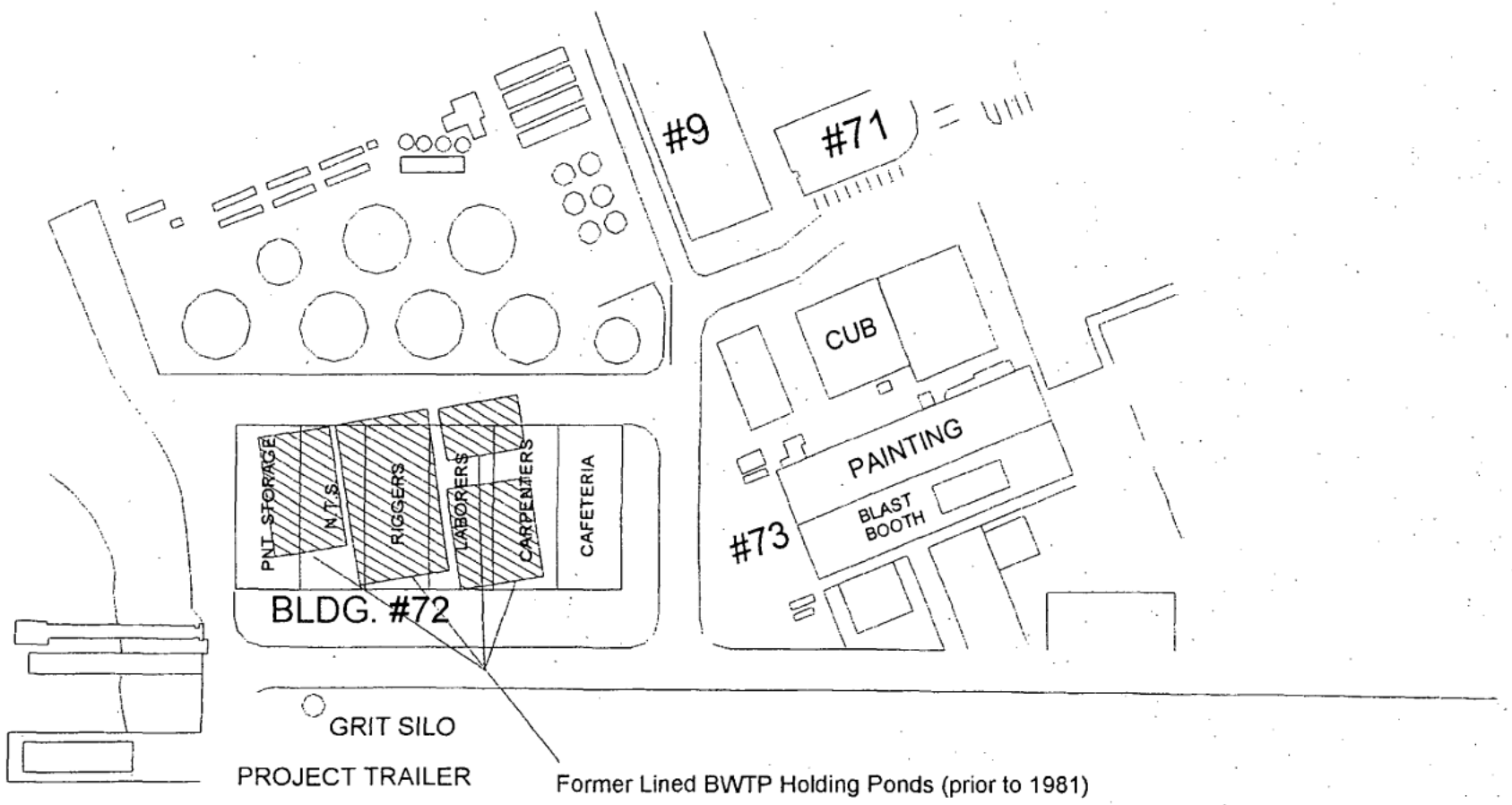


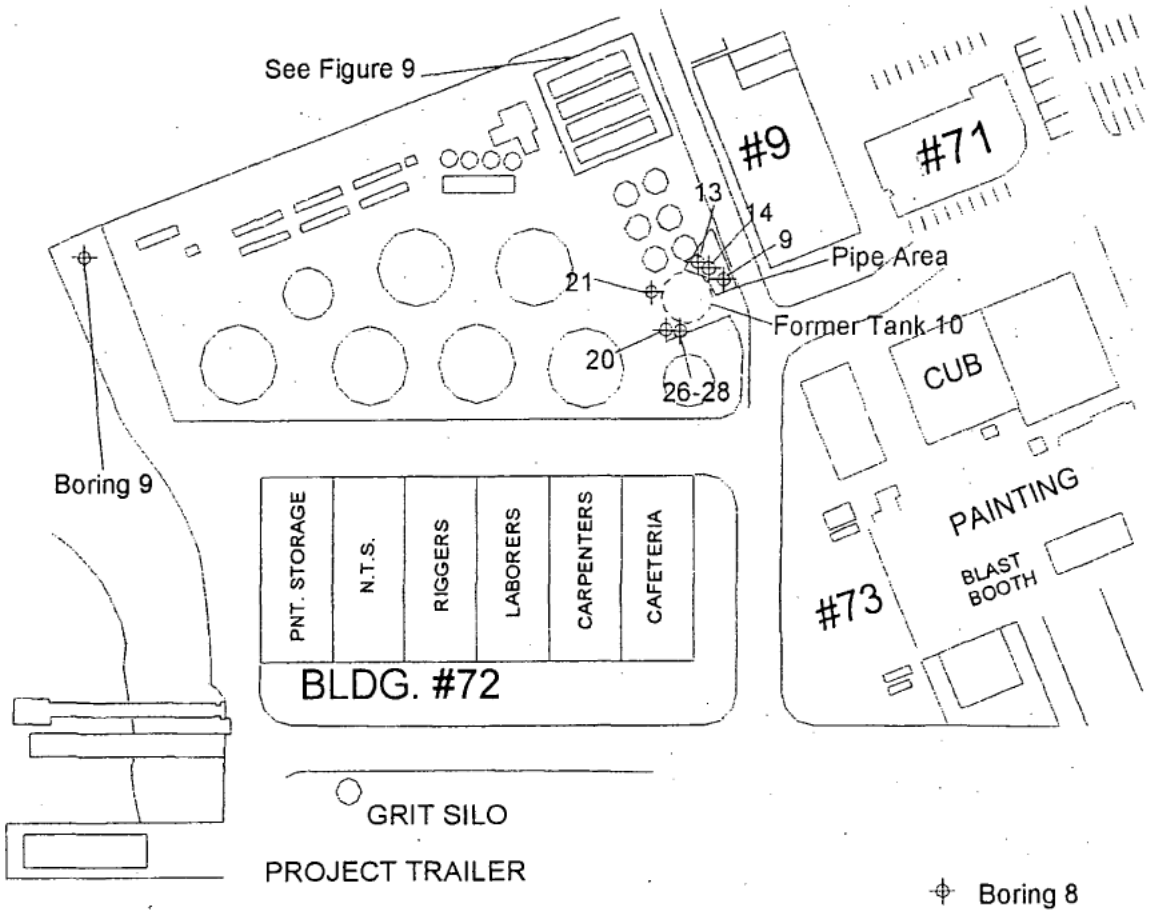
Figure 6
Generalized North-South Cross Section
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 7
Former BWTP Pond Locations
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Legend:

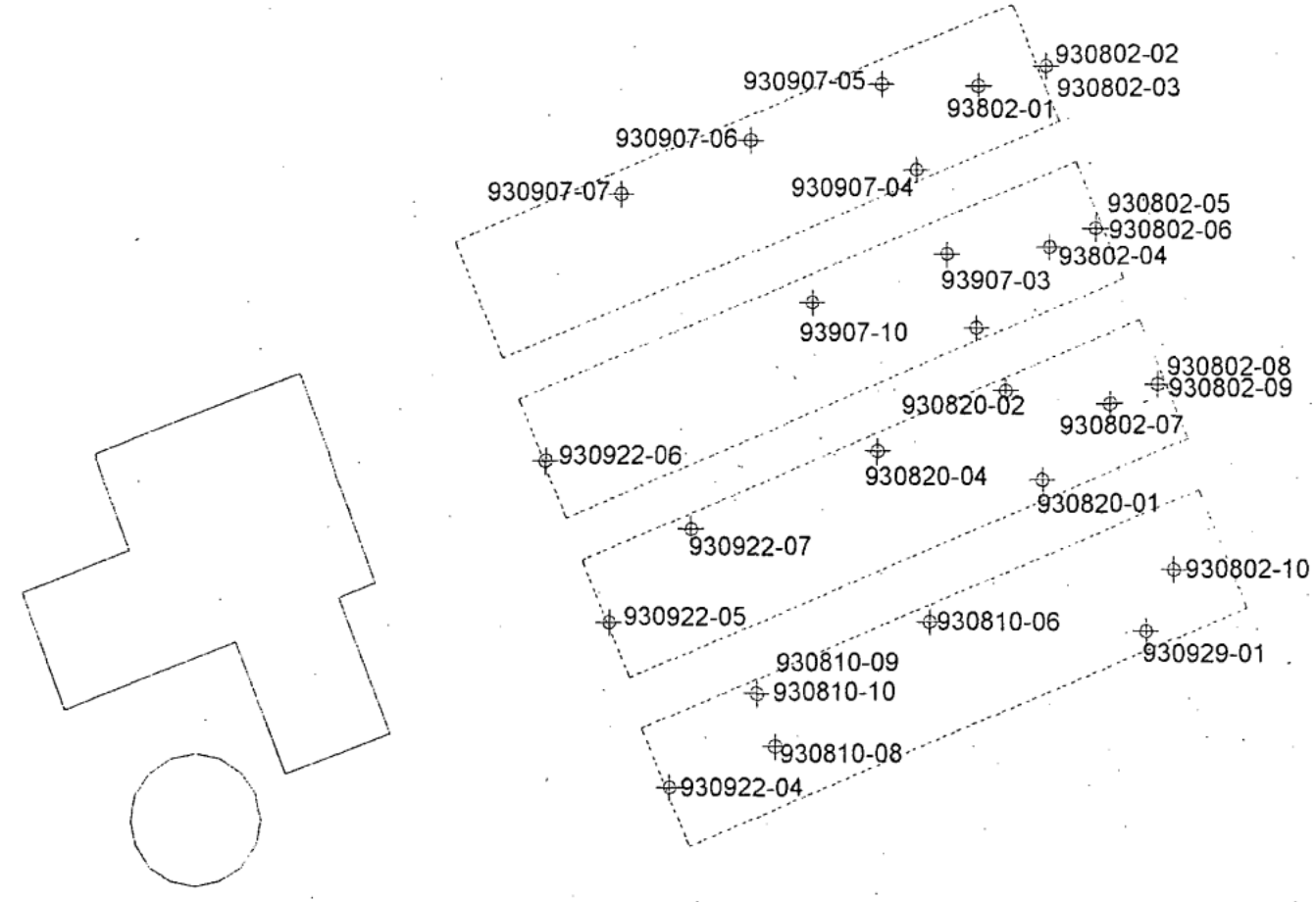
⊕ Port and Cascade General Soil Investigation, 1998

⊕ Tank 10 and Pipe Area Investigation, 1993

0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 8
Past Sampling Locations - BWTP Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



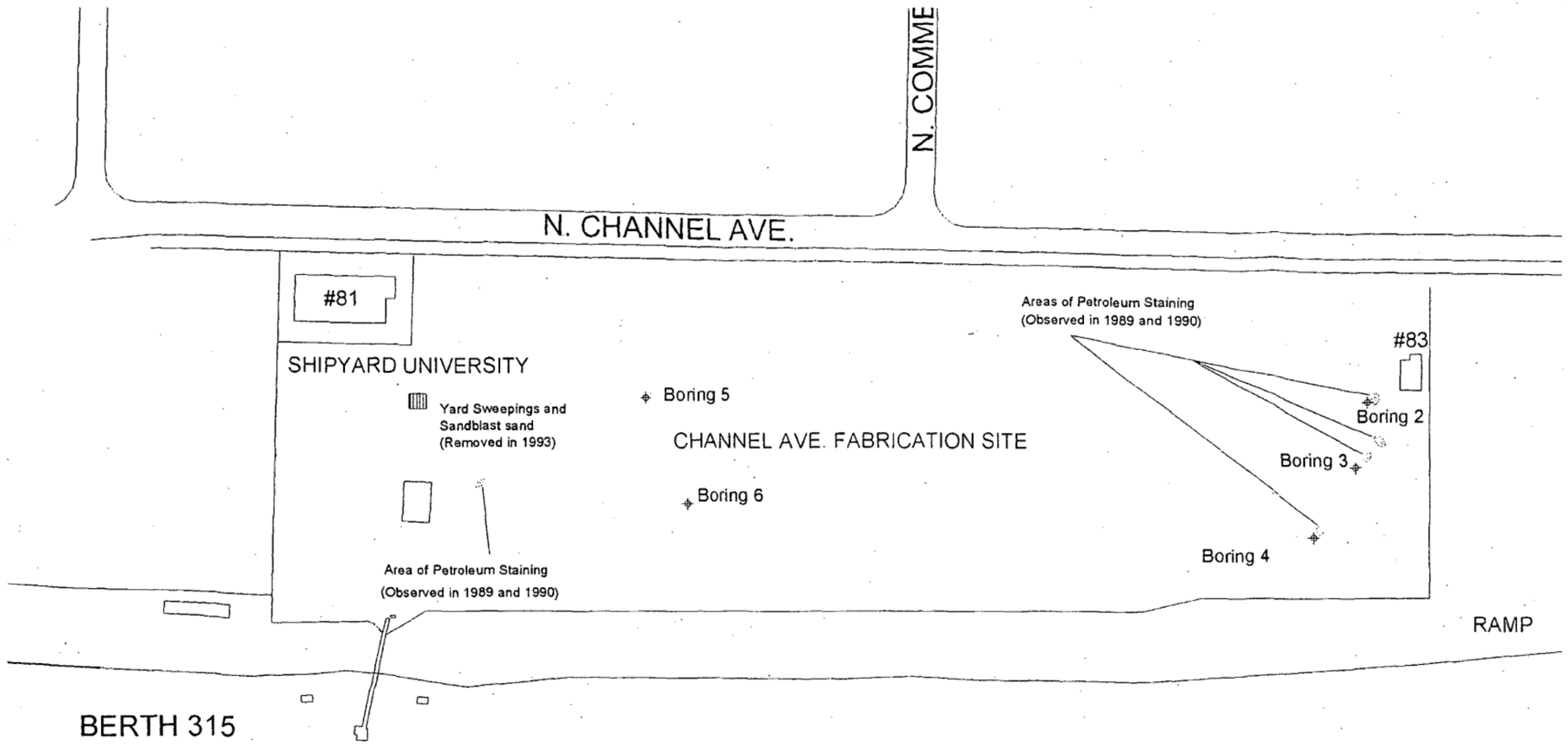
Legend:

⊕ BWTP Level II Environmental Assessment Confirmation Sampling Locations, 1994

0 ft. 10 ft. 20 ft. 40 ft.

N

Figure 9
Past Sampling Locations - BWTP ASTs
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



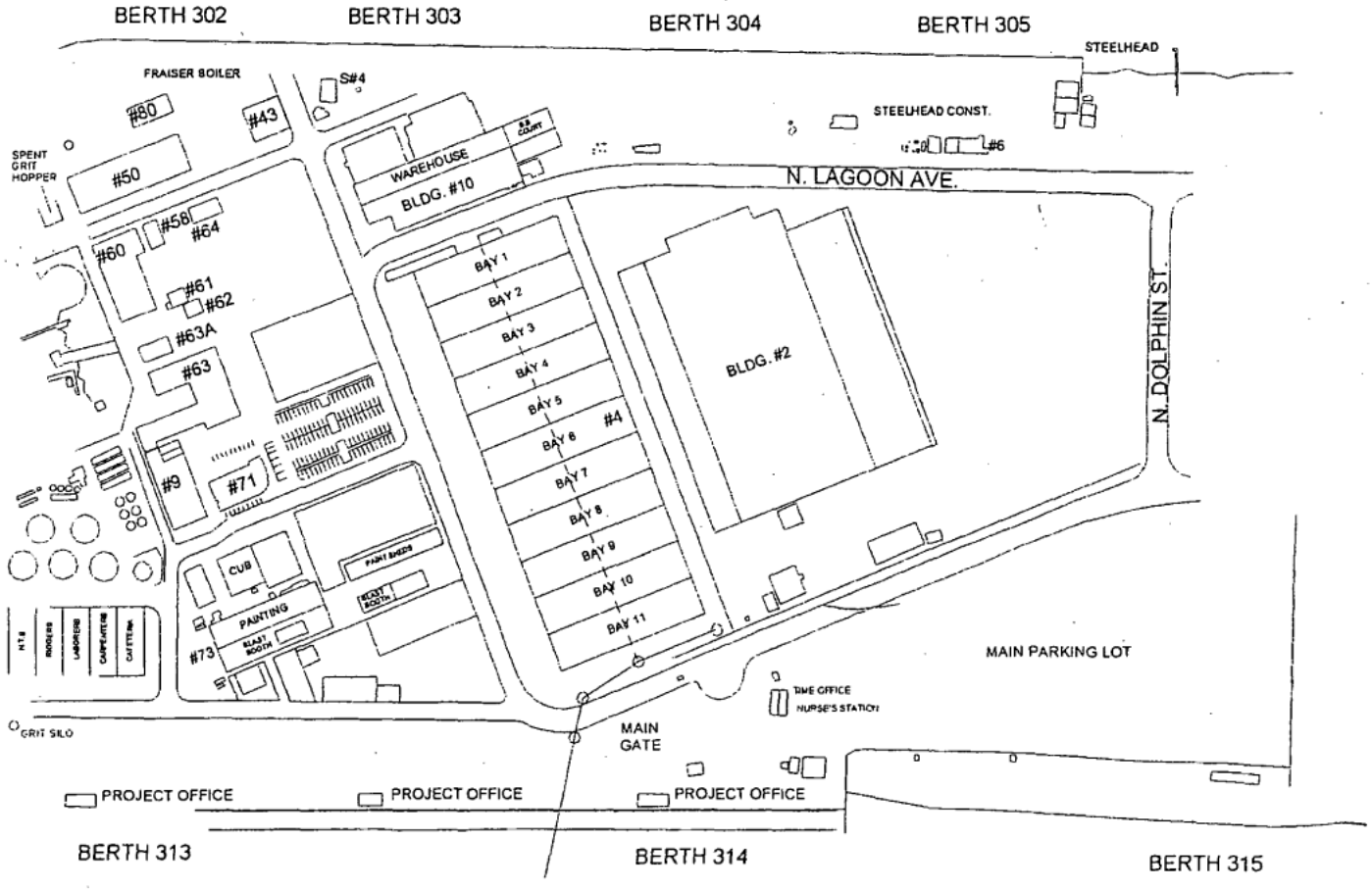
Legend:

⊕ Port and Cascade General Soil Investigation, 1998

0 ft. 100 ft. 200 ft. 400 ft.

N

Figure 10
Past Sampling Locations - N. Channel Avenue Fabrication Site
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



0 ft. 200 ft. 400 ft. 800 ft.

N

Figure 11
Building 4 Floor Drain Outfall Location Map
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

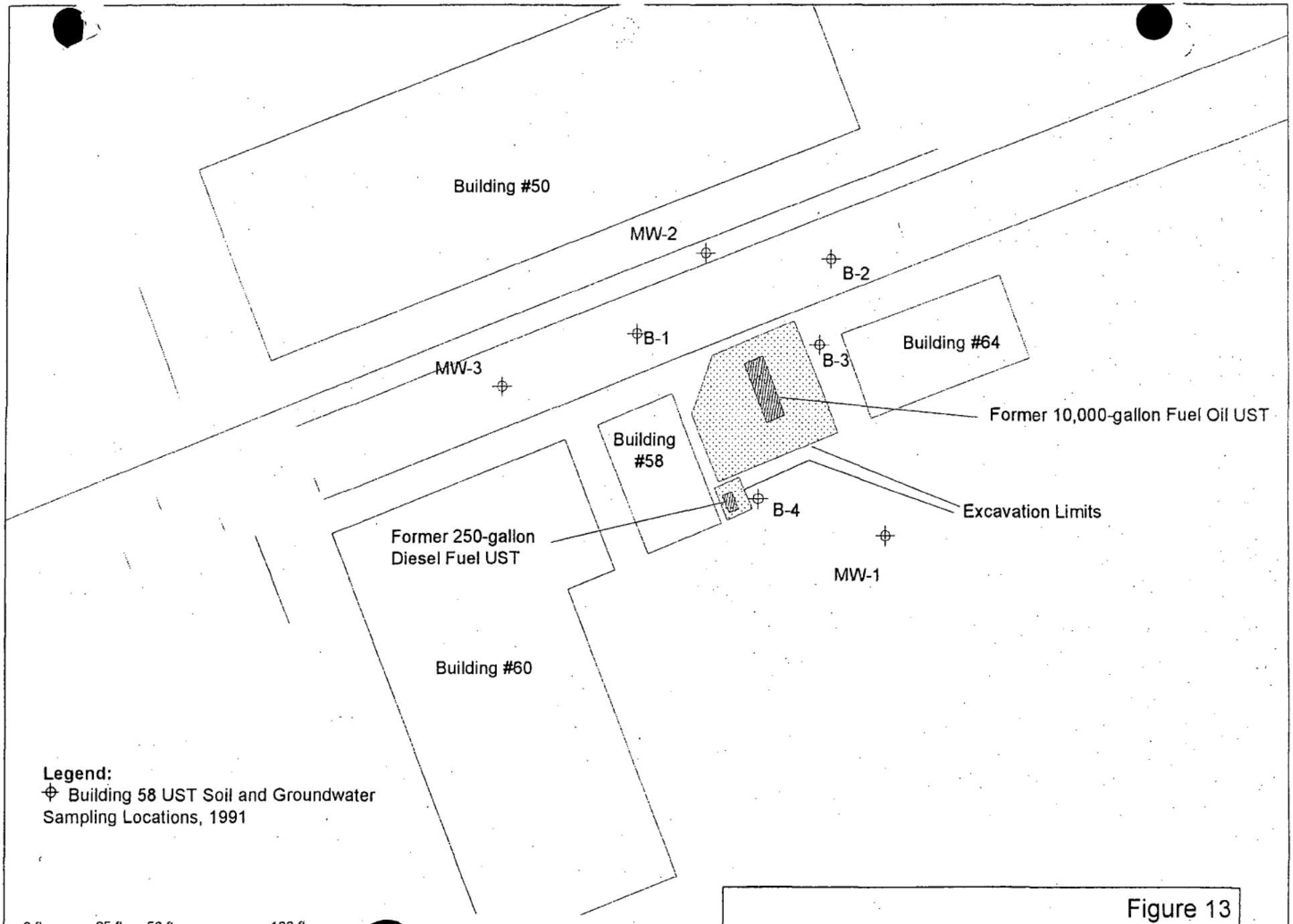
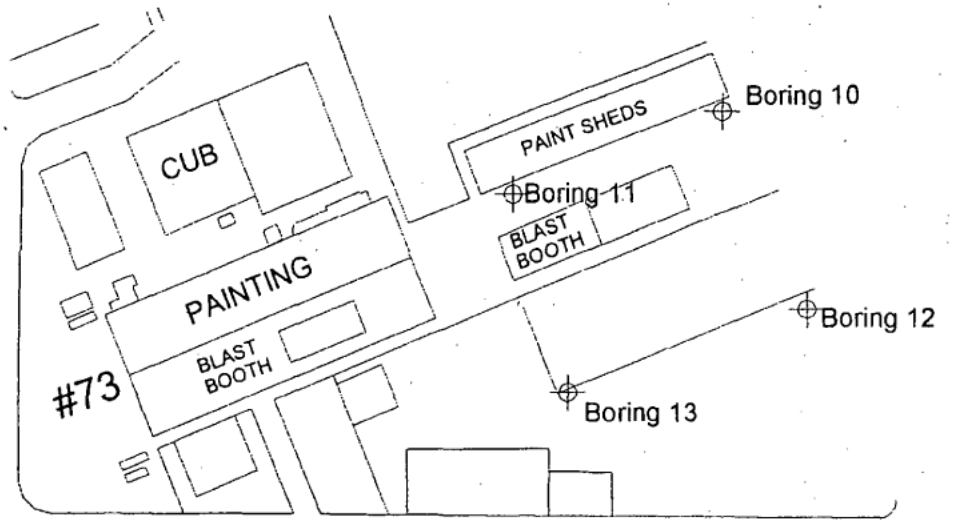


Figure 13
Past Sampling Locations - Building 58 UST
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Boring 8

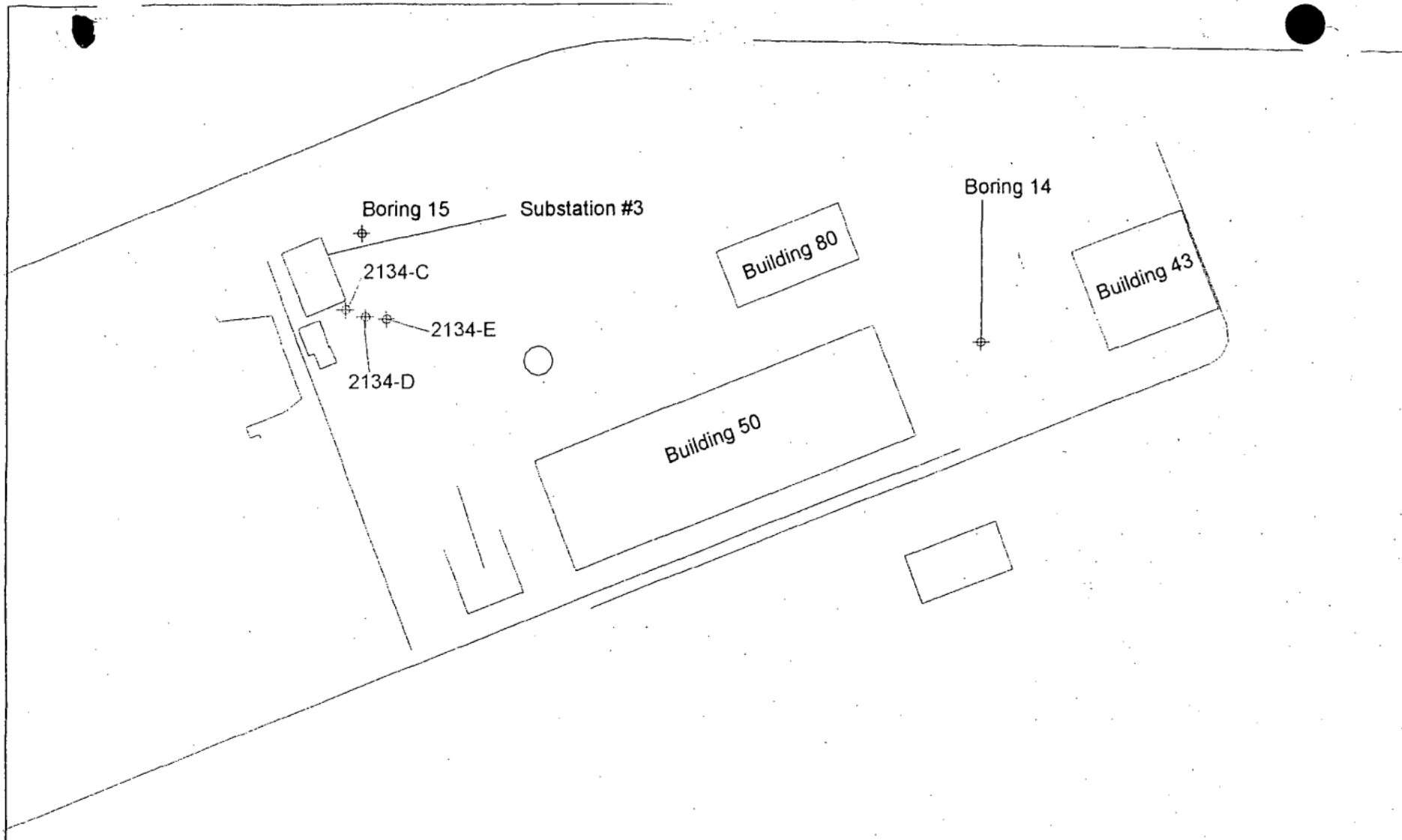
Legend:

Port and Cascade General Soil Investigation, 1998

0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 14
Past Sampling Locations - Paint Shed/ Blast Booth Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Legend:

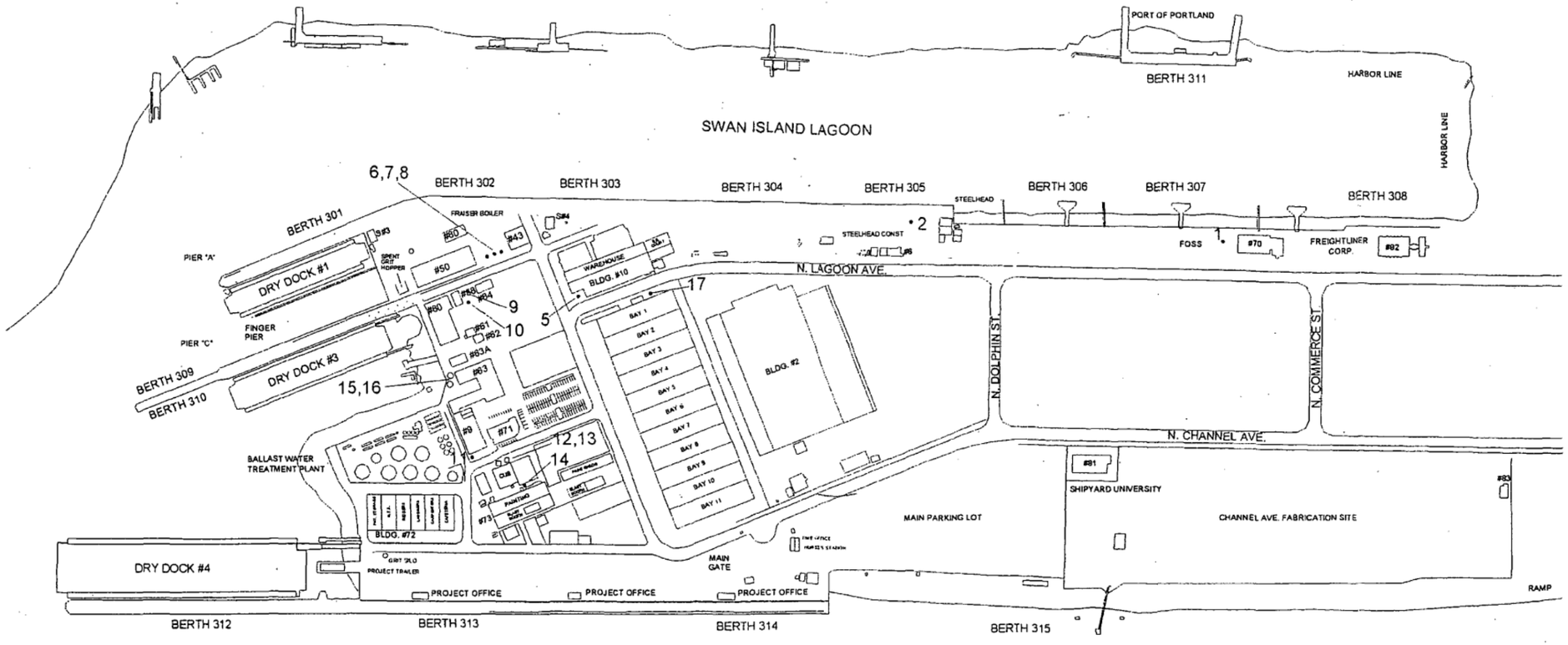
⊕ Port and Cascade General Soil Investigation, 1998

⊕ Stoddard Solvent and Diesel/Oil Cleanup Verification Sample Locations, 1992

0 ft. 50 ft. 100 ft. 200 ft.

N

Figure 15
Past Sampling Locations - Building 43, 50, 80 and Substation 3 Areas
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



WILLAMETTE RIVER

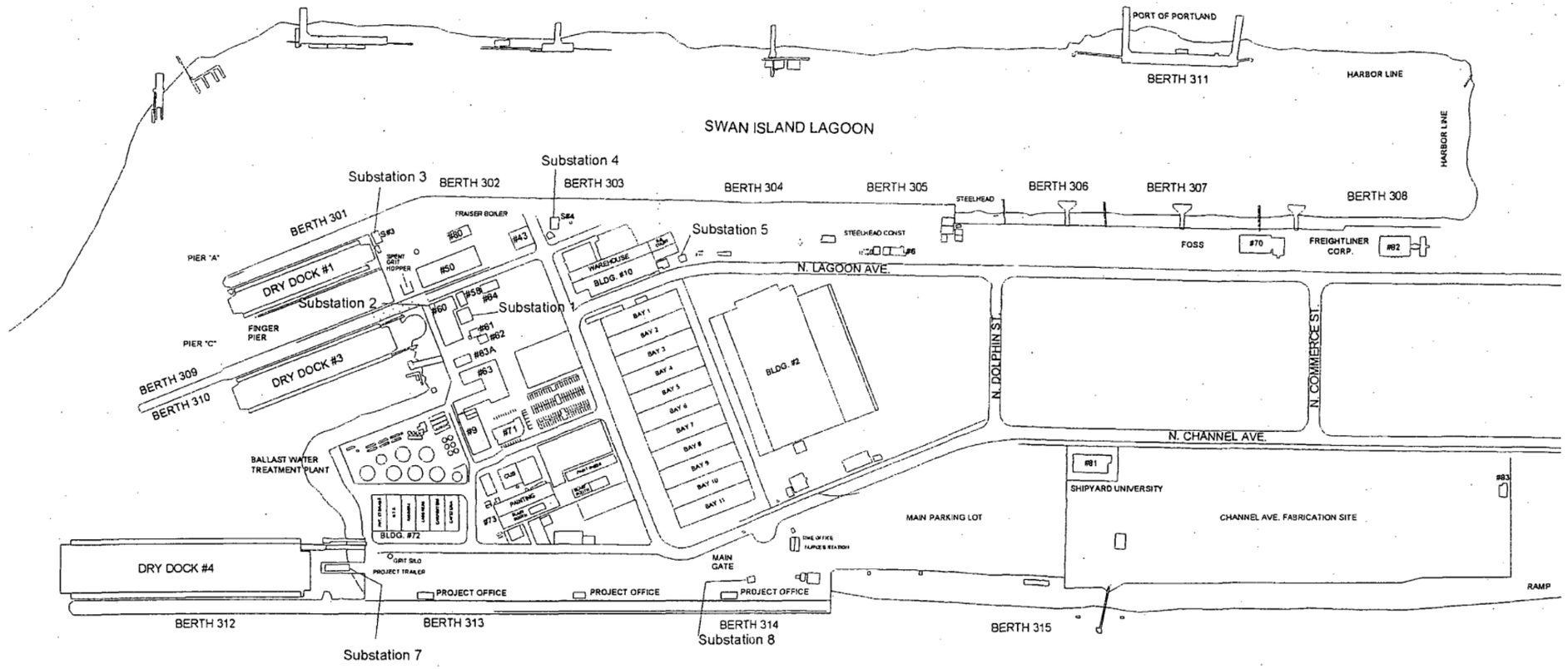
Legend:

- • Former UST Location
- ○ Current UST Location
- ▫ Current HOT Location

0 ft. 250 ft. 500 ft. 1000 ft.

N

Figure 12
Former and Current UST and HOT Location Map
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



WILLAMETTE RIVER

0 ft. 250 ft. 500 ft. 1000 ft.

N

Figure 16
Substation Location Map
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

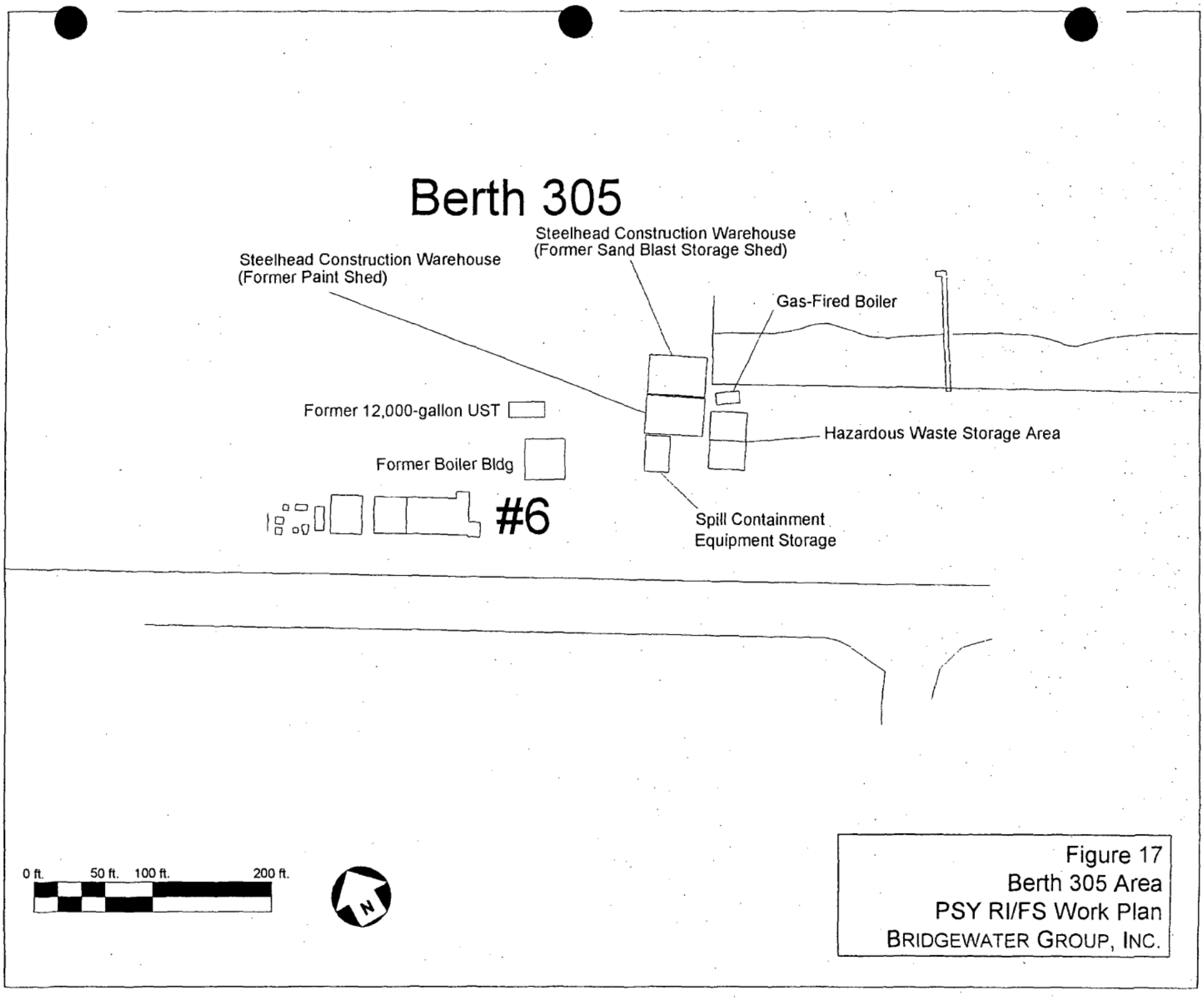
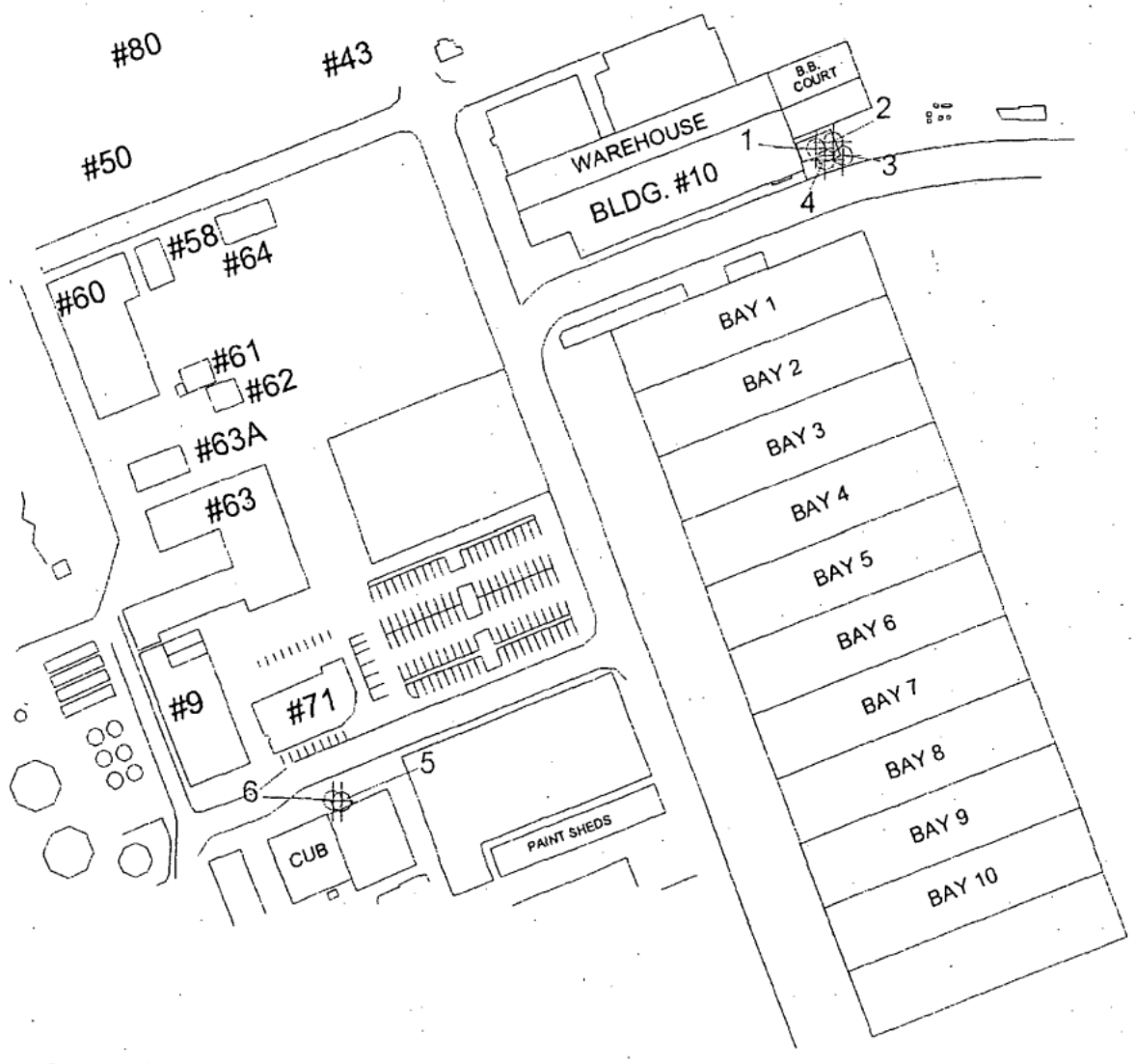


Figure 17
Berth 305 Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



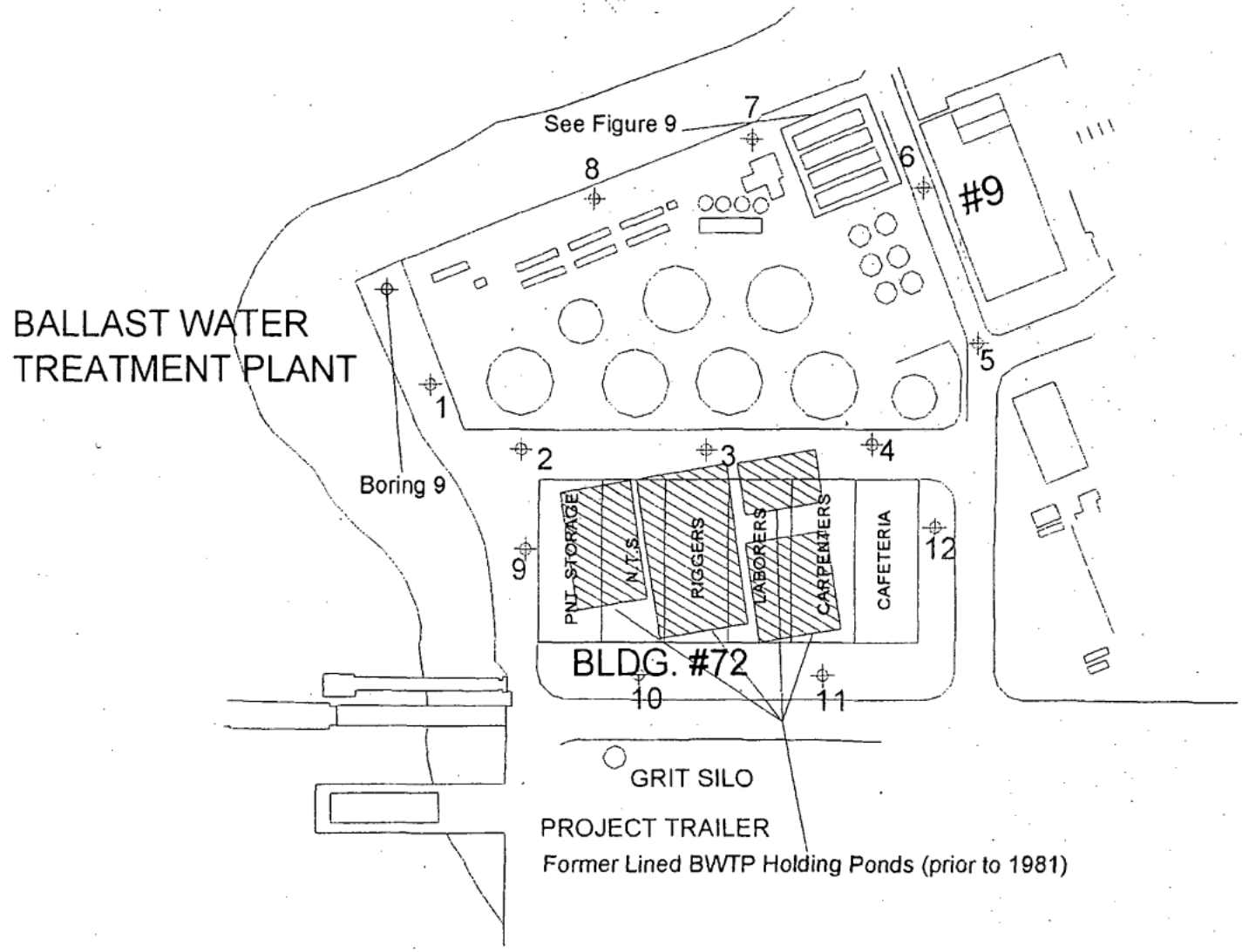
Legend:

⊕ Norvac Hazardous Material Storage Area Investigation, 1992

0 ft. 100 ft. 200 ft. 400 ft.

N

Figure 18
Past Sampling Locations - Former Norvac Hazardous Materials Storage Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Legend:
⊕ Phase IA RI Push-Probe Locations
⊕ Port and Cascade General Soil Investigation, 1998

0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 19
Push-Probe Locations - BWTP Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

N. CHANNEL AVE.

#81

SHIPYARD UNIVERSITY

Yard Sweepings and Sandblast sand (Removed in 1993)

26
25
27

Area of Petroleum Staining (Observed in 1989 and 1990)

Boring 5

CHANNEL AVE. FABRICATION SITE

14
13 Boring 6
15
16

Areas of Petroleum Staining (Observed in 1989 and 1990)

#83

Boring 2
22
23
Boring 3
21
27
17
18
19
Boring 4
20

RAMP

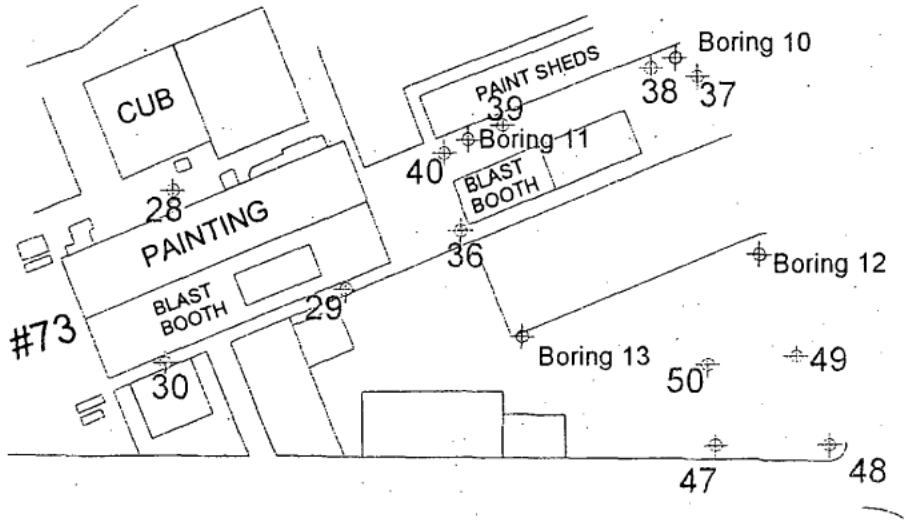
BERTH 315

Legend:
- Phase IA RI Push-Probe Locations
- Port and Cascade General Soil Investigation, 1998

0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 20
Push-Probe Locations - N. Channel Avenue Avenue Fabrication Site
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



Legend:
Phase IA RI Push-Probe Locations
Port and Cascade General Soil Investigation, 1998

o ft. 75 ft. 150 ft. 300 ft.

N

Figure 21
Push-Probe Locations - Building 73, Paint Shed/Blast Booth Area, and Former Hazardous Waste Storage Area
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

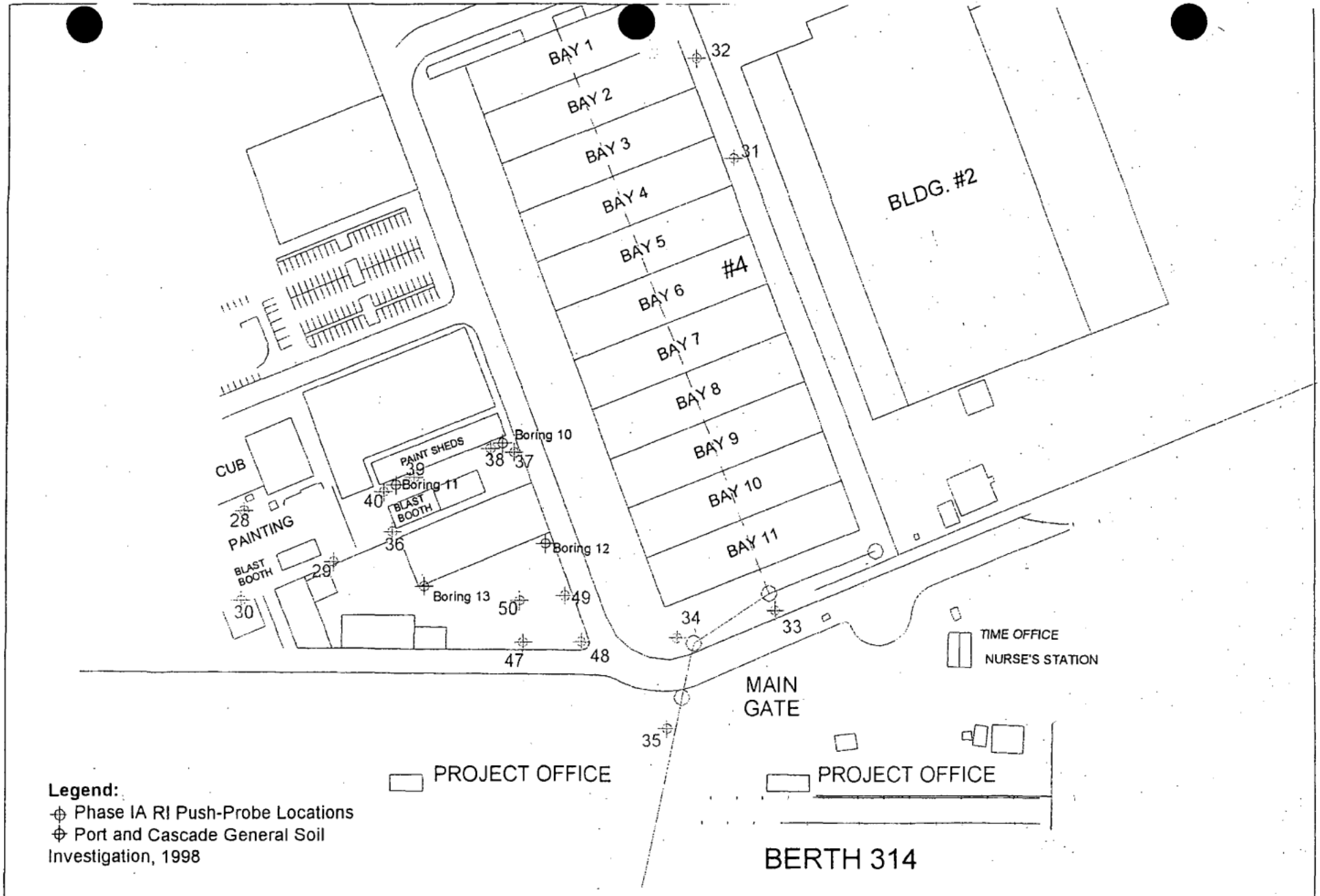
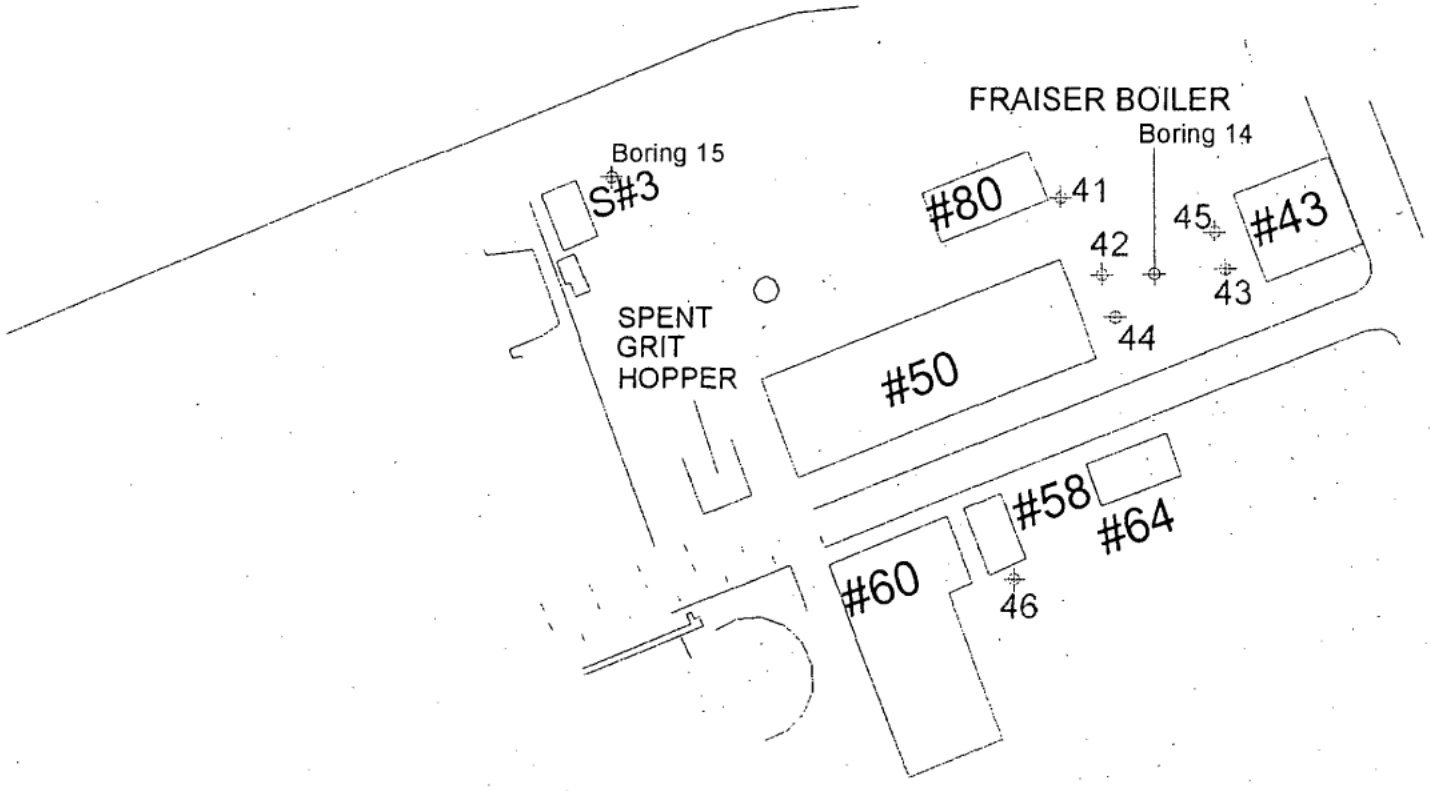


Figure 22
Push-Probe Locations - Building 4
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

BERTH 302

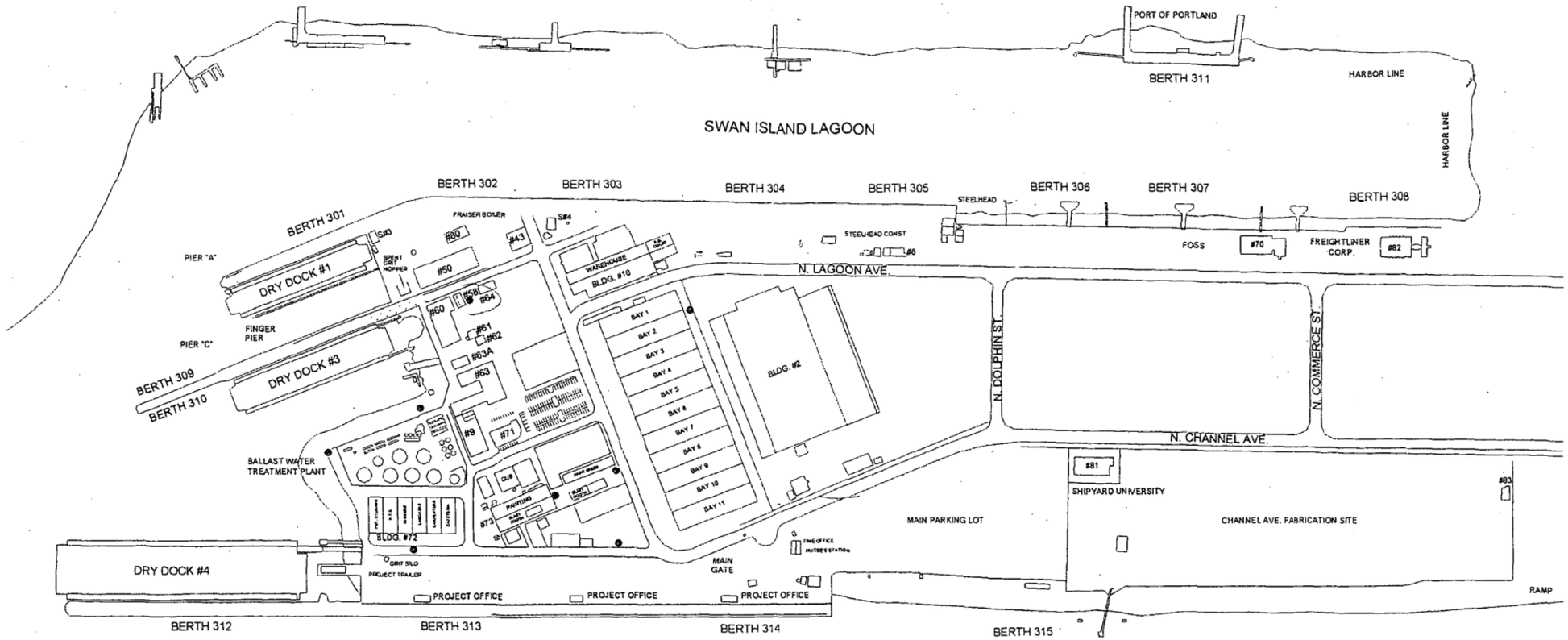


Legend:
- Phase IA RI Push-Probe Locations
- Port and Cascade General Soil Investigation, 1998

0 ft. 75 ft. 150 ft. 300 ft.

N

Figure 23
Push-Probe Locations - Old Boiler and Building 43, 50, 80 and 43 Areas
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



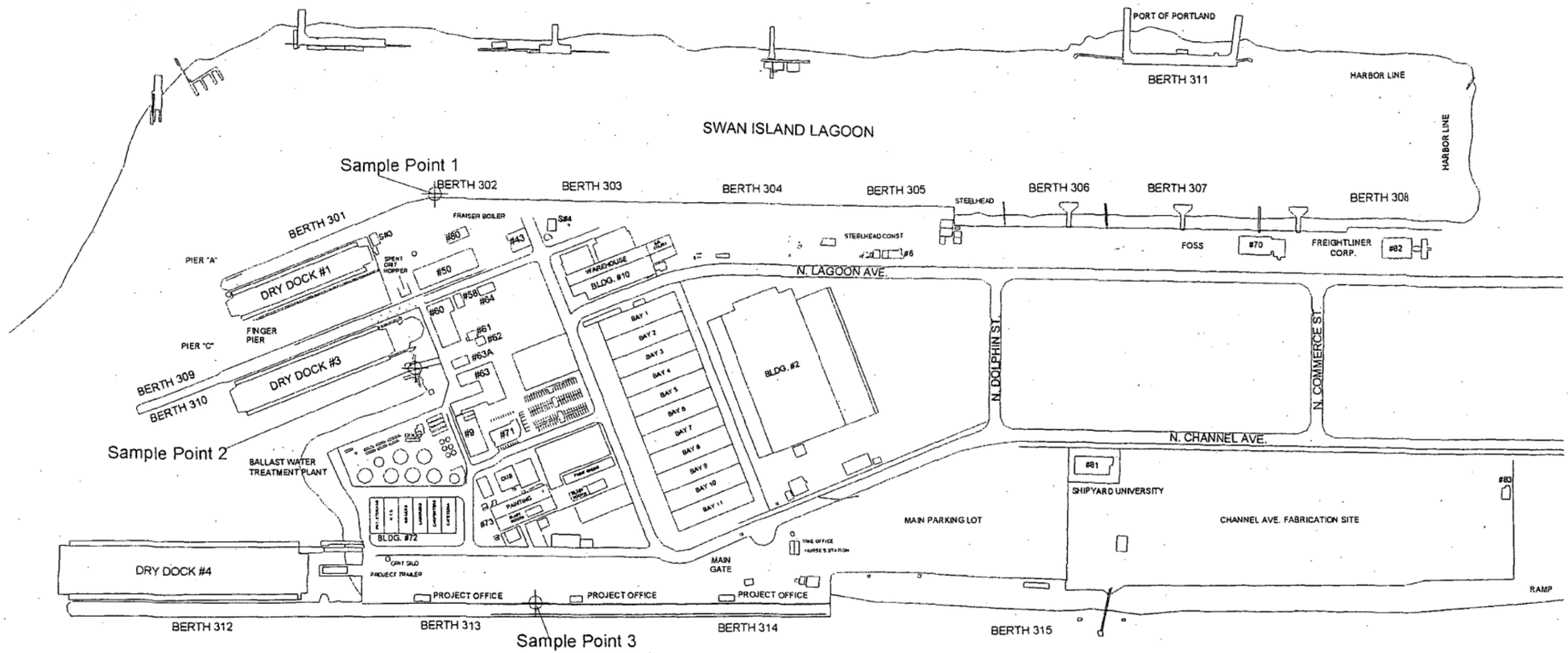
WILLAMETTE RIVER

Legend:
● Phase IB Groundwater Monitoring Well Locations

0 ft. 250 ft. 500 ft. 1000 ft.

N

Figure 24
Groundwater Monitoring Well Locations
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.



WILLAMETTE RIVER

Legend:

⊕ Cascade General Storm Water Sampling Locations

0 ft. 250 ft. 500 ft. 1000 ft.

N

Figure 25
Cascade General Storm Water Monitoring Locations
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

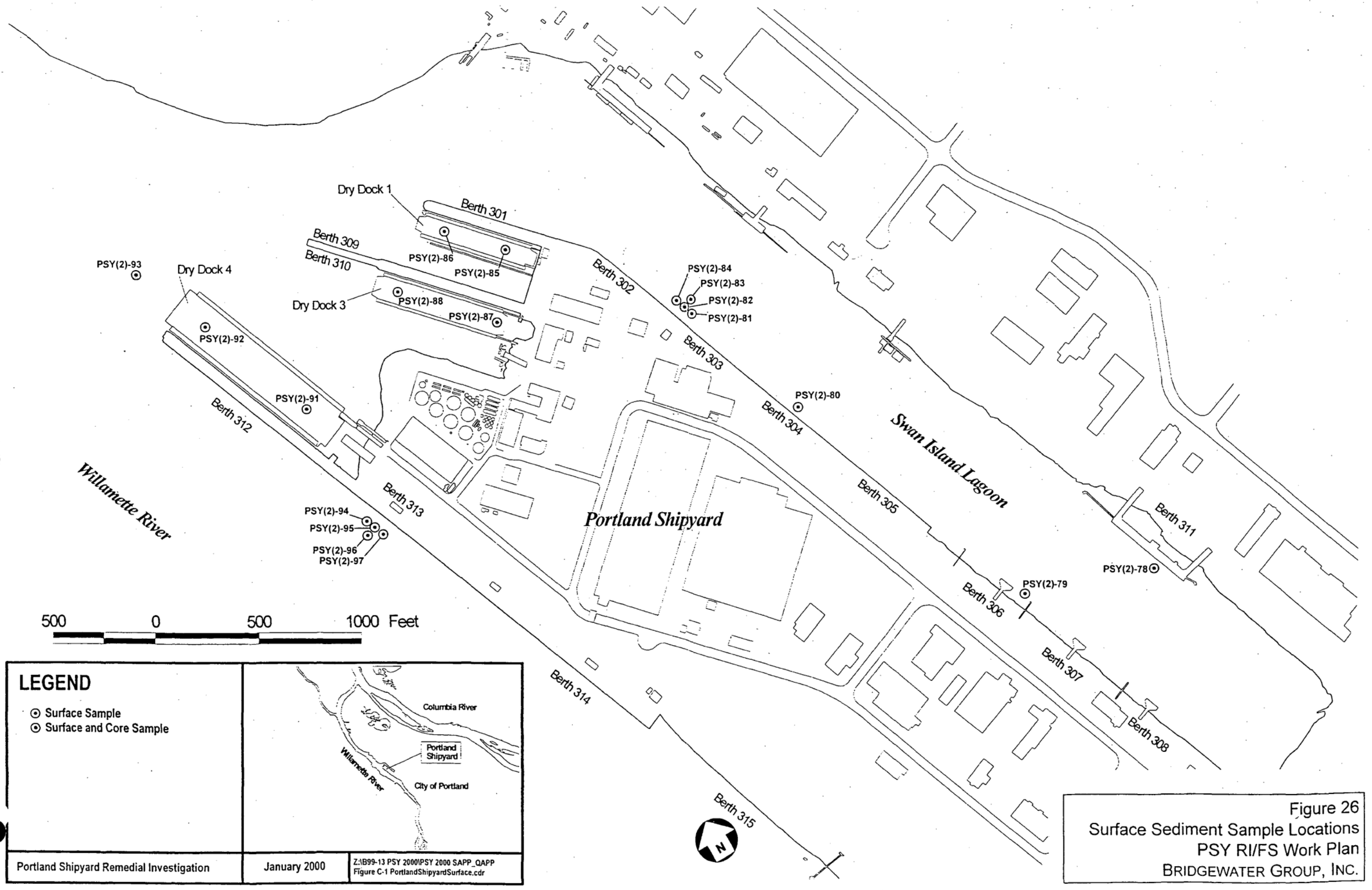
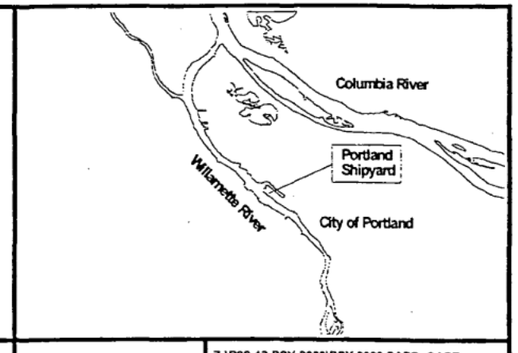


Figure 26
Surface Sediment Sample Locations
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

Dry Dock 1
Berth 301
PSY(2)-85
Berth 309
Berth 310
Berth 302
PSY(2)-93
Dry Dock 4
PSY(2)-83
PSY(2)-82
Dry Dock 3
PSY(2)-87
Berth 303
Swan Island Lagoon
PSY(2)-89
PSY(2)-90
Berth 304
PSY(2)-91
Berth 312
Willamette River
Berth 313
Portland Shipyard
Berth 305
Berth 311
PSY(2)-78
Berth 306
Berth 307
Berth 308
Berth 314
Berth 315
N

LEGEND

- ⊙ Core Sample
- ⊙ Surface and Core Sample



Portland Shipyard Remedial Investigation | January 2000 | Z:\B99-13 PSY 2000\PSY 2000 SAPP_QAPP Figure C-2 PortlandShipyardSubsurface.cdr

Figure 27
Subsurface Sediment Sample Locations
PSY RI/FS Work Plan
BRIDGEWATER GROUP, INC.

APPENDIX A – PROJECT MANAGEMENT PLAN

APPENDIX A – PROJECT MANAGEMENT PLAN

Introduction

This Project Management Plan (PMP) describes the roles of key personnel involved in the PSY RI/FS project, reporting requirements, procedures for handling variations from the Work Plan, and a proposed schedule of submittals and RI/FS activities.

Project Site Description

The PSY address is 5555 North Central Channel Avenue in Multnomah County, Portland, Oregon. The PSY is located in north Portland, between Swan Island Lagoon and the Willamette River on the peninsula known as Swan Island.

The Site includes 94 acres of uplands and 106 acres of submerged lands. The Site also includes Berth 311, a concrete pier/lay berth located on the east side of Swan Island Lagoon.

Current operations at the PSY consist of the repair and maintenance of privately owned and government vessels from the United States and overseas. Ship repair and maintenance are conducted in three dry docks (i.e., Dry Docks 1, 3 and 4) and 15 berths along the perimeter areas of the shipyard. The shipyard's upland areas, or yard, house the support services for both ship repair operations and maintenance of the shipyard infrastructure.

Associated Documents

This PMP was developed in conjunction with, and is accompanied and supplemented by, other documents, all prepared according to agency guidelines. Accompanying documents are as follows:

Work Plan – A document that presents the scope of work for the RI/FS

Appendices B and C – Sampling and Analysis Plans (SAPs) for soil and groundwater and for surface water and sediments. A SAP is a detailed description of the scope of the RI and procedures that will be used to collect samples and complete chemical analyses. It includes quality assurance/quality control (QA/QC) procedures for both the field and laboratory. The SAP is intended to serve as a manual for field staff. Appendix B contains the SAP for upland sampling activities. Appendix C contains that SAP for surface water and sediment sampling activities.

Appendices D and E – Health and Safety Plans (HSPs) for Hahn and Associates and Striplin Environmental Associates. A HSP presents a description of procedures to be used in the field to protect personnel from potential hazards that may exist during on-site activities.

Project Management and Organization

The following is an outline of the roles of key personnel involved in the project. Key personnel are also identified. Should any changes in key personnel occur during the course of the project, the DEQ will be notified in writing.

Project Manager

The project manager's primary responsibilities will be to oversee and coordinate the activities of the technical consultant and to serve as the point of contact with the DEQ and the technical consultant. The project manager is:

Trey Harbert
Port of Portland
P.O. Box
Portland, Oregon 97208
Phone: (503) 944-7325
FAX: (503) 944-7353
Email: harbet@portptld.com

Technical Consultant Project Manager

The technical consultant project manager will have responsibility for overseeing the activities associated with the Portland Shipyard (PSY) RI/FS. He will be responsible for the overall management of the project and coordination of the project team and will be the point of contact for all communications directed at the technical consultant. Additional responsibilities of the project manager include: schedule control and adjustments; cost control and reporting; and identification of potential problems and the development of contingency plans to respond to the identified problems. The technical consultant project manager is:

Stuart M. Brown
Bridgewater Group, Inc.
4640 SW Macadam Avenue, Suite 222
Portland, Oregon 97201
Phone: (503) 973-6068
FAX: (503) 973-6069
Email: sbrown@bridgeh2o.com

A resume for Stuart Brown is included at the end of this appendix.

Subcontractors

Several activities will be completed with the aid of subcontractors. Not all of the subcontractors have been identified. Listed below are the activities that will be subcontracted and the currently identified subcontractors.

- Upland sampling: Hahn and Associates
- Surface water and sediment sampling: Striplin Environmental Associates
- Drilling and well construction: GeoTech, Inc.
- Sediment sampling boat, grab sampler, and vibracore: Marine Sampling Services (MSS)
- Chemical analytical laboratory: Columbia Analytical Services
- Biological testing laboratory: Northwestern Aquatic Sciences

Reporting Requirements

Reporting requirements include monthly progress reports, RI report, risk assessment report, and FS report. To the extent practicable, reports will be duplex printed on recycled paper. Except for monthly progress reports, all reports will be submitted as drafts for review and comment. Upon incorporation of applicable comments, the report will be produced in final form.

Report distribution will be as follows:

Port of Portland: 5 copies all reports; 4 bound and 1 unbound

DEQ: Monthly Reports: 2 copies

All other reports: 2 bound; 1 unbound

Monthly Reports

Monthly reports will be submitted to the DEQ by the 10th day of the month following the reporting period. The monthly reports will summarize activities performed, data results collected or received, and problems encountered or resolved during the past month and activities planned for the upcoming two months.

Remedial Investigation Report

The RI report will be consistent with the suggested outline presented in EPA's "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA." The RI report will include the following:

- Executive Summary
- Introduction (purpose and report organization)

- Site Background (including current and reasonably likely future land and water uses)
- Study Area Investigation
- Summary and Conclusions
- Appendices

Risk Assessment Report

The results of the ecological and human health risk assessment will be presented in the Ecological and Human Health Risk Assessment Report. Specifically, the report will cover the following major topics:

- Identification of Chemicals of Concern
- Exposure Model
- Exposure Assessment
- Toxicity Assessment
- Human Health and Ecological Risk Characterization
- Uncertainties

Feasibility Study Report

The results of the FS will be presented in the Feasibility Study Report. The outline of the report will be consistent with the suggested outline presented in EPA's "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" and will include requirements specific to Oregon's environmental cleanup program as described in DEQ's "Final Guidance for Conducting Feasibility Studies." Specifically, the report will cover the following major topics:

- Introduction
- Identification and Screening of Technologies
- Development and Screening of Alternatives
- Detailed Analysis of Alternatives
- Recommendation of the Remedial Action

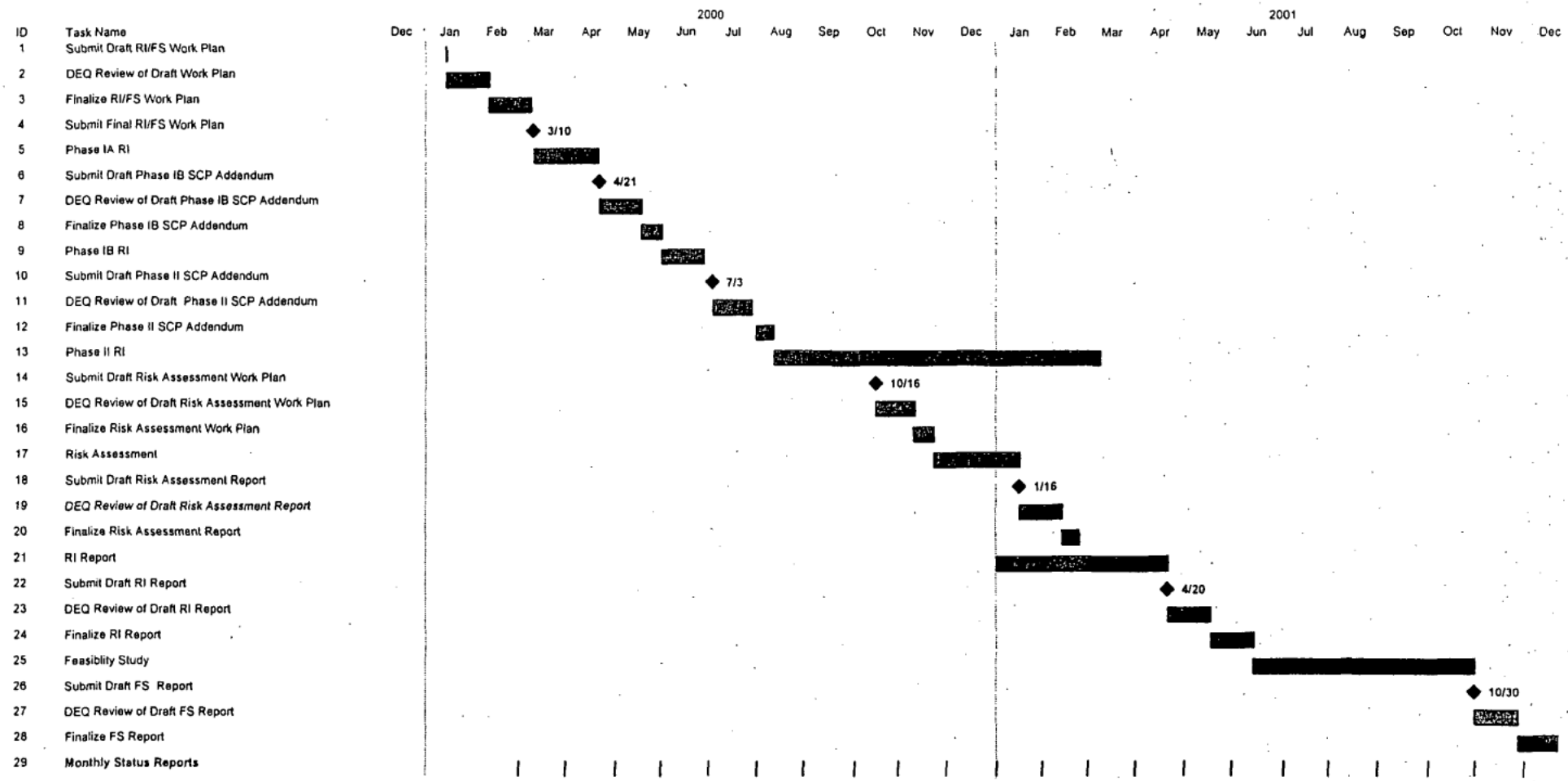
Project Schedule

Figure A-1 provides a schedule for the RI/FS. This schedule presents target dates for planning purposes only. The schedule will be adjusted as necessary.

DEQ will be notified in writing of all subsequent modifications to the project schedule, should they occur. The notice shall include an explanation and justification of the changes.

DEQ will be notified at least 5 working days in advance of RI sampling activities.

Figure A-1
Project Schedule
PSY RI/FS



Project: PSYRIFSWorkPlan
Date: Tue 1/11/00

Task | Progress | Summary | Rolled Up Split | Rolled Up Progress | Project Summary
Split | Milestone | Rolled Up Task | Rolled Up Milestone | External Tasks

STUART M. BROWN

President

Education

M.S., Civil Engineering, Stanford University - 1976
B.S.E., Civil Engineering, Arizona State University - 1975

Professional Registrations

Civil Engineering, State of Washington, No. 19431

Summary of Relevant Experience

In April of 1998, Mr. Brown joined with three other leading environmental consultants to form Bridgewater Group, Inc.

For the Port of Portland, Mr. Brown reviewed the available literature on river hydraulics, the transport of sediments, and the quality of sediments in the Portland Harbor. His work was used by the Port of Portland as the technical foundation for the Port's contaminated sediment management strategy. His work was also used by DEQ in the preparation of the Portland Harbor Sediment Management Plan.

Also for the Port of Portland, Mr. Brown is currently serving as the Portland Shipyard (PSY) RI/FS project manager under DEQ's voluntary cleanup program. To date, Mr. Brown has assisted the Port of Portland in reviewing and commenting on DEQ's strategy recommendation and file review memorandum for the PSY. He has also prepared a detailed evaluation of the dredging and dredged material disposal history of the PSY. As the PSY RI/FS project manager, Mr. Brown assisted the Port of Portland in their review of Cascade General's proposal to conduct ship dismantling for the U.S. Navy. Mr. Brown's work included a review of environmental issues associated with past Navy ship dismantling activities and a review of Cascade General's environmental operations plan for the proposed ship dismantling project.

Finally, for the Port of Portland, Mr. Brown has provided technical review of DEQ's Portland Harbor Sediment Management Plan, specifically in the areas of site discovery; sediment and contaminant transport in the Portland Harbor; and alternative approaches to establishing sediment quality guidelines.

Mr. Brown recently served as a senior technical consultant to Portland General Electric on the cleanup of the Bors site, a former electrical transformer recycling and copper recovery operation. This cleanup project is being conducted under DEQ's voluntary cleanup program using the PCB generic remedy. Mr. Brown reviewed soil sampling investigation results for PCBs, lead, arsenic, and dioxins/furans; he assisted Portland General Electric in the evaluation of hot spots and remedial alternatives; and he provided oversight during the excavation and land disposal of contaminated soils.

Mr. Brown provided strategic advice to a group of potentially responsible parties seeking to cleanup a site under Oregon's new generic remedy for PCB sites. Mr. Brown worked with utilities in Oregon and the DEQ to jointly develop this generic remedy. He is providing the PRP group with advice on how to keep the site within the definition of a "PCB site" so that the cost of cleanup can be dramatically reduced.

From 1984 to 1998, Mr. Brown worked for CH2M HILL in Portland and Seattle, Washington. During his tenure with CH2M HILL, he held a number of senior management positions. In addition, he directed a number of relevant projects for clients in Oregon and Washington:

- For Portland General Electric, he managed the first remediation project completed under Oregon's original site cleanup law. The project involved the design and implementation of a remedial action that involved low-volume dredging, water treatment, sediment disposal, and capping of PCB-contaminated sediments in the Willamette River. This $1,000,000 project was completed on time and below the originally estimated cost. For PGE, he also managed the remediation of upland soils containing heavy metals, PCBs, and petroleum hydrocarbons at the same site.

- For Weyerhauser, Mr. Brown managed a $300,000 remedial investigation/feasibility study for Weyerheuser's Longview Mill Chlor-Alkali facility. By following an innovative strategy of proactive investigation and cleanup, Mr. Brown was able to successfully reduce the Washington State Department of Ecology's level of concern about the site and defer Weyerhaeuser's need to additional action for 5 years. His project strategy also deferred the need to investigate Columbia River sediments that may contain mercury from the facility. This outcome significantly reduced Weyerhaeuser's short-term costs by $200,000 to $500,000.

During the period of 1983 to 1984, Mr. Brown was a Vice President for Anderson-Nichols in Palo Alto, California. He was responsible for the firm's groundwater and hazardous waste practices. Major accomplishments included successful completion of an Electric Power Research Institute project that demonstrated the limited risks posed by PCB releases from utility electric equipment and a study to develop one of the first guidance documents under the EPA Superfund program.

During the period of 1976 to 1983, he managed the Hydrologic Systems Section for Battelle, Pacific Northwest Laboratories in Richland, Washington. His organization consisted of a group of 40 surface water and groundwater hydrologists that provided technical support to and direction of waste management projects for the Department of Energy at the Hanford Reservation, Nuclear Regulatory Commission, Environmental Protection Agency, and private industry. In this role, Mr. Brown managed numerous projects related to sediment transport and the associated transport of pesticides, heavy metals, and radionuclides in aquatic systems.

APPENDIX B – UPLAND SAMPLING AND ANALYSIS PLAN

UPLAND
SAMPLING AND ANALYSIS PLAN

Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

December 17, 1999

Prepared for:

Bridgewater Group, Inc.
Portland, Oregon

Prepared by:

Hahn and Associates, Inc.
Portland, Oregon

Project No. 4800

TABLE OF CONTENTS

ATTACHMENT
B-1 Example Field Logs and Forms

UPLAND
SAMPLING AND ANALYSIS PLAN

Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

December 17, 1999

1. INTRODUCTION

The Upland Sampling and Analysis Plan (SAP) is designed to cover all phases of remedial investigation (RI) activities conducted at the site. Because the extent of subsequent phases, if necessary, is not known, this SAP includes a more comprehensive description of procedures and sampling methodologies than necessary for use as part of the Phase I RI activities. A more comprehensive SAP was developed in an effort to streamline subsequent phases to the RI process. Procedures and/or methodologies proposed for use that are not included in this SAP, will be submitted in addendum form to the Oregon Department of Environmental Quality (DEQ).

2. INVESTIGATION METHODS

A number of investigative methods may be used to collect soil, sediment, storm water, groundwater, and air samples at the subject site. Surface soil samples will be collected directly or by slide-hammer methodology. Subsurface soil samples will be collected from drilled soil borings, push probe borings, and/or hand auger borings. Screening-level groundwater samples will be collected from either temporary well points installed in drilled soil borings, or from drive points installed in push probe borings. If storm water and/or air sampling become necessary during subsequent phases of investigation, sampling procedures and methodologies will be included in an addendum to this SAP.

Soil samples will typically be collected at pre-determined locations that are chosen based on available information, as described in the SCP. These samples will be used to identify the presence of chemicals in known or suspected source areas and/or to determine the nature and extent of contamination. In cases where a statistical sampling strategy is warranted, such as to collect data for risk assessment purposes, a systematic grid methodology will be utilized to establish a statistically-valid soil sample population (see Section 3.2).

All investigation activities, where applicable, will be conducted in accordance with the Oregon Groundwater Law (Oregon Revised Statute (ORS) Chapter 537) and the Rules for Construction and Maintenance of Monitoring Wells and Other Holes in Oregon (Oregon Administrative Rules (OAR) Chapter 690, Division 240).

2.1 Soil Boring Installation Procedures

Soil borings will be installed by several methods depending on soil type and equipment access. Typically, the first preference for soil boring installation will be by push probe techniques. In areas where soil types are not appropriate for push-probe methodology (i.e. in some gravelly or cemented soils), or in cases where monitoring wells are to be installed, drilled borings can be installed by any number of methods. In areas where access is limited, where underground utilities are present, and/or where only shallow soil samples are required, hand auger borings will be installed.

2.1.1 Push-Probe Soil Borings

A truck-mounted push probe unit that uses a 1.5-inch outside-diameter (OD) hydraulically-driven steel rod will be used to advance the proposed push probe soil borings. Soil and/or screening-level groundwater samples may be collected from these borings.

2.1.2 Drilled Soil Borings

In areas where soil types are not appropriate for push-probe methodology (i.e. in some gravelly or cemented soils), or in cases where monitoring wells are to be installed, drilled borings can be installed by several methods. The soil types and proposed depths of investigation at the site are appropriate for use of hollow-stem auger drilling methodology. As such, drilled soil borings will be installed with a hollow stem auger drilling rig equipped with 4 1/4-inch inside-diameter (ID) auger. Soil borings with monitoring well installations will be completed with a hollow stem auger drilling rig equipped with 6 3/4-inch ID auger. The suitability of hollow stem auger borings will be evaluated prior to use in situations where dense non-aqueous phase liquids (DNAPLs) are expected and/or encountered.

2.1.3 Hand Auger Soil Borings

Hand-augured soil borings will be installed with a stainless-steel hand auger with 2-inch OD hollow bit.

2.2 Soil Boring Abandonment Procedures

Following installation, each drilled boring, push probe, or hand auger boring will be backfilled with granular bentonite from the bottom of the hole to land surface. For borings with temporary well points, the well point will be pulled out of the hole and the sand pack drilled out prior to abandonment. For boreholes with more than 25 feet of standing water, a grout-slurry mixture placed by tremie pipe will be used to backfill the borehole.

In areas of asphalt or concrete surface, the borings will be backfilled with bentonite chips to within 1.5 feet below ground surface (bgs) and capped with concrete to the land surface.

2.3 Monitoring Well Installation and Development

All monitoring well installation activities will be conducted in accordance with the Oregon Groundwater Law (Oregon Revised Statute (ORS) Chapter 537) and the Rules for Construction and Maintenance of Monitoring Wells and Other Holes in Oregon (Oregon Administrative Rules (OAR) Chapter 690, Division 240).

All monitoring wells will be installed through 6 3/4-inch ID hollow stem augers. The monitoring wells will be constructed with 2-inch ID, threaded, schedule 40, PVC blank casing and slotted screen. Typically, ten (10) feet of 0.010-inch slotted screen will be set at the bottom of each well with blank casing extending to the ground surface. Actual monitoring well screen depths and intervals are discussed in the SCP.

A sand pack will be placed in the annular space from the bottom of the borehole to 3 feet above the top of the screen with a Colorado 10/20 silica sand. The wells will then be developed with a surge block to set the sand pack. In shallow wells (typically up to 40 feet bgs), a well seal composed of 3/8-inch bentonite chips will be placed on top of the sand pack to a depth of about 2 feet bgs and hydrated.

For wells with over 25 feet of standing water in the annular space, the use of a grout-slurry mixture is required by the Oregon Water Resources Division (OWRD) in OAR 690-240-005 through 180. In the case of the deep-zone wells, a two-foot bentonite plug will be placed on top of the sand pack. The well seal, composed of a cement-bentonite slurry, will then be placed by a tremie pipe from the top of the bentonite plug to the within two feet of land surface. Deep-zone wells are not proposed as part of the Phase I SCP.

In general, monitoring wells will be completed with flush well monuments cemented in at the surface. However, there may be instances where above-ground monuments will be advantageous, and as such, their suitability will be evaluated while in the field. Above-ground monuments also require the installation of three surrounding guard posts. The well casings will be fitted with locking caps.

At least 24 hours following installation of the monitoring wells, they will be further developed by purging with a submersible or peristaltic pump in an attempt to remove the fine sediment from around the well bore. During development, at least 10 well volumes of water, and the volume of any well construction water, will be removed from each well. The parameters pH, temperature, and conductivity will be measured during the development process. Following purging, the wells will be considered developed when the parameters of pH, temperature, and conductivity have stabilized. Stabilization is considered to have been met when the last three measured values for each of the above parameters are within 10 percent of each other.

2.4 Field Measurements

Field instruments are used to screen for organic vapors, to measure water for parameters that help ensure collection of representative groundwater samples, and to measure water levels and detect the presence of non-aqueous phase liquids (NAPLs) in wells.

A photoionization detector (PID) will be used to screen the breathing zone during drilling activities for health and safety purposes, as well as to screen for headspace vapors from collected soils samples (see Section 3.4). Organic vapor concentration measurements will be conducted utilizing a MicroTIP Model MP-1000 equipped with a PID and a 10.6 electron volt (eV) lamp.

During purging of wells prior to sample collection, the temperature, pH, and conductivity of the water is measured to monitor for stabilization. The temperature, pH, and conductivity will be measured with a Hydac model probe.

If necessary, turbidity and dissolved oxygen can also be measured on water samples in the field. Turbidity will be measured with a LaMotts Model 2008 turbidity meter. Dissolved oxygen will be measured with a YSI Incorporated Model 55 dissolved oxygen meter.

Water level measurements will be taken with a Solinst water level indicator (conductive probe). Measurements for the presence and thickness of NAPL will be taken with a Waterra HS-I hydrocarbon interface sensor.

2.5 Analytical Methods and Procedures

Constituents of potential concern at the site include petroleum hydrocarbons, VOCs including BTEX, SVOCs including PAHs, PCBs, and metals. All analytical methods will follow standard U.S. Environmental Protection Agency (EPA) procedures as outlined in *Test Methods for Evaluating Solid Wastes - Physical/Chemical Methods* (SW-846) as updated and DEQ-approved methods where necessary.

The analytical methods expected for use during this project are as follows:

Parameter	Analytical Method	
	Soil	Water
Hydrocarbon Identification	NW TPH-HCID	NW TPH-HCID
Gasoline-Range TPH	NW TPH-G	NW TPH-G
Diesel-Range TPH	NW TPH-Dx	NW TPH-Dx
Oil-Range TPH	NW TPH-Dx	NW TPH-Dx
VOCs, including BTEX	EPA 8260B	EPA 8260B
BTEX	EPA 8021B	EPA 8021B
SVOCs, including PAHs	EPA 8270C	EPA 8270C
PAHs	EPA 8270 SIM	EPA 8270 SIM

PCBs	EPA 8082	EPA 8082
Total Metals		
Arsenic, Cadmium, Chromium		
Copper, Nickel, Lead, Tin, Zinc	EPA 6010B/7000	EPA 200.8
Mercury	EPA 7470A	EPA 245.1

2.6 Land Surveying

All soil borings and monitoring wells will be surveyed for location and elevation to City of Portland datum by an Oregon Registered Professional Land Surveyor. For monitoring wells, both the ground surface and the top of the casing elevations will be surveyed. All survey data will be collected within an accuracy of 0.01 feet vertically and 0.1 feet horizontally. Relevant physical features will also be surveyed in order to compile an accurate map of the study area. However, legal property boundaries will not be surveyed.

2.7 Aquifer Slug Tests

Aquifer slug tests will be performed on selected monitoring wells at the site to gather data on aquifer characteristics (hydraulic conductivity). Hydraulic conductivity estimates will allow for an estimation of groundwater flow velocity and the rate of contaminant migration, if any, in the groundwater, as well as the design of pumping wells, if necessary.

Rising-head slug tests will be performed by lowering a PVC slug into the well to displace the groundwater in the well. The displaced water in the well is then allowed to return to the original static water level and the slug is quickly removed from the well. This procedure simulates the instantaneous removal of a slug of water from the well. The water level recovery data is measured by a pressure transducer and recorded with an electronic data logger. The recovery data will be analyzed by the Bouwer and Rice, 1976[1] solution method to calculate preliminary estimates of transmissivity.

The number and location of aquifer slug tests to be conducted will be discussed in the Phase II SCP addendum. Aquifer pumping tests are not proposed as part of the Phase II RI.

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[1] Bouwer, H. and R. C. Rice, *A Slug Test for Determining Hydraulic Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells*, Water Resources Research, 12 (1976), pg 423-428.

3. SOIL SAMPLING PROCEDURES

3.1 Soil Sample Collection Procedures

Soil samples may be collected via any of the following methods which are described in more detail below: subsurface soils from drilled soil borings, push probe borings, or hand auger borings, from surface soils directly by trowel or via a slide-hammer sampler, and solids from catch basins or manholes. Soil sample locations will typically be selected using a biased sampling strategy (i.e. based on available information), although a statistical sampling strategy using systematic grid methodology (Section 3.2) may also be utilized.

Upon collection, all samples will be labeled and transferred to a chilled container for shipment to the analytical laboratory. Standard sampling protocols, including the use of chain-of-custody documentation, will be followed for all sampling procedures as discussed in Section 7.5.

3.1.1 Drilled Soil Borings

Soil samples from drilled borings will be collected with a 2-inch OD split-barrel sampling device that will be driven into the undisturbed soils 1.5 feet ahead of the drill bit, using a Standard Penetration Test (SPT). In cases where volatile compounds are being investigated, soil samples will be collected by fitting the sampling device with 6-inch long, brass sleeves.

Soil samples collected from the split-spoon sampling device or hand auger bit will be manually transferred to a 9-ounce sample jar and capped with Teflon lined lid. In cases where brass sleeves are utilized (i.e., where volatile compounds are of interest), the sleeve containing the sample will be capped with Teflon paper, plastic end caps, and sealed with non-volatile silicon tape.

Drilled soil borings are not proposed as part of the Phase I RI. If proposed for use, drilled boring soil samples will be collected at pre-determined intervals in accordance with a DEQ-approved Work Plan addendum, which may be modified based on field screening indicators (discussed in Section 3.9).

3.1.2 Push Probe Borings

In push probe borings, continuous soil cores will be collected using a 2-inch OD, 4-foot long, stainless-steel, Macro-Core sampler, fitted with a polyvinyl chloride (PVC) sleeve, that will be advanced 4 feet into the undisturbed soils.

Soil samples will be collected from push probes by cutting open the PVC sleeve and placing soil in a 9 ounce sample jar that is capped with Teflon lined lid. In cases where volatile compounds will be analyzed, soil samples will be collected by cutting a 6-inch long section from the PVC sleeve that will immediately be capped with Teflon paper, plastic end caps, and sealed with silicon (non-VOC) tape.

Soil samples will be collected from the cores at pre-determined depth intervals, as outlined in the SCP, as well as from other depths based on field screening indicators (discussed in Section 3.9).

3.1.3 Hand Auger Borings

In cases where access is an issue, soil samples may be collected from a hand auger equipped with a 2-inch OD hollow bit. Once the desired sampling depth is attained, the hand auger will be removed from the hole and soil collected from the auger tip.

In cases where volatile compounds will be analyzed, samples will be collected with a decontaminated slide-hammer sampler equipped with a 6-inch long brass sleeve. Once the desired sampling depth is reached by the hand auger equipment, it will be removed whereupon the slide-hammer device will be used to drive the brass sleeve 6-inches into the undisturbed soils.

3.1.4 Surface Soil Samples

Surface soil samples will be collected by scraping approximately 3 inches of soil from the surface or immediately beneath asphalt or concrete surface cover and sampling soils with a decontaminated stainless-steel trowel. In cases where the surface sample will be analyzed for volatile compounds, a slide-hammer sampling device will be used to drive a brass sleeve 6 inches into the undisturbed soils. Surface soil sample locations are discussed in the SCP.

3.1.5 Manhole/Catch Basin Solids Samples

Samples of solids from manholes or catch basins will be collected from the inside bottom portion of manholes/catch basins with a decontaminated stainless-steel hand auger. For safety reasons, manholes will not be physically entered by sampling personnel. The solids samples will be collected at the bottom portion of the installation in areas away from the main flow. Manhole/catch basin samples are not proposed as part of the Phase I RI. If proposed for use, samples will be collected in accordance with a DEQ-approved Work Plan addendum.

3.2 Grid-Based Sampling

In areas where little background information is available or where a statistically-significant population of soil samples is desired, then a grid-based sampling methodology will be utilized. In these cases, sample locations will be chosen based on a systematic random grid sampling procedure in accordance with U.S. Environmental Protection Agency (EPA) protocol, as defined in *Test Methods for the Evaluation of Solid Wastes, (SW-846), Physical*

Chemical Methods, Third Edition, Volume II, dated November 1986. These protocols are designed to generate data which is representative of the areas of concern. The protocol requires that a sufficient number of samples be collected that represent the variability of the concentrations of contaminants. Grid-based sampling is not proposed for use during the Phase I RI.

3.3 Soil Sample Description

The properties of all soil samples will be noted in the field by an HAI scientist/geologist. The properties of the soil, including color, moisture, plasticity, grading of coarse-grained soils, and texture, will be noted in the field and incorporated into a boring log for each subsurface boring. An estimate of the Unified Soil Classification System (USCS) soil type designation (ASTM D 2487-85) will also be shown on the boring logs. The USCS soil type designation will be a field estimate only and will not be confirmed by laboratory analyses. Visual or olfactory evidence of contaminant occurrence in the samples, if present, will also be noted on the boring logs as discussed in Section 3.9.

3.4 Soil Field Screening Procedures

All soil samples will be screened in the field for the presence of contaminants by visual (color), olfactory, sheen, and headspace vapor methods. Selected soil samples may be screened in the field for the presence of NAPLs by the ultraviolet fluorescence method. The results of the field screening observations will be noted on the field boring log.

The presence of sheen will be assessed by placing clean tap water in a black pan and introducing approximately 5 grams of disaggregated soil to the water. The observations for the presence or lack of sheen is a relative indicator of contamination.

Organic vapor levels in the soil samples will be measured by the headspace vapor method utilizing a MicroTIP Model MP-1000 equipped with a PID and a 10.6 electron volt (eV) lamp. Immediately following the collection of the sample, approximately 4 ounces of soil will be placed in a one-quart plastic bag and sealed. The sample will then be set aside for an approximate 20-minute stabilization period where the sample is allowed to reach ambient temperature. The detector probe is then inserted through the seal into the bag to collect the headspace sample. The results of the headspace screening will be recorded on the boring log in parts per million (ppm). The results of the headspace method will be used for qualitative screening purposes only.

Where necessary, the presence of non-aqueous phase liquids (NAPLs) will be screened for in the field using ultraviolet (UV) fluorescence equipment. A Raytech view box and light equipped to provide short-wave (2,500 to 3,000 angstrom units) and long-wave (3,000 to 4,000 angstrom units) UV light will be utilized. Following the collection of each soil sample, approximately 2 ounces of soil will be placed in the view box. The short-wave UV light will be activated and the sample will be observed for fluorescence. Subsequently, the short-wave

UV light will be deactivated, the long wave UV light will be activated, and again the sample will be observed.

4. GROUNDWATER SAMPLING PROCEDURES

4.1 Screening-Level Groundwater Samples from Temporary Well Points

Screening-level groundwater samples from drilled borings will be collected from temporary well points that are installed in the borehole. When using hollow stem auger drilling methods, vertical profiling of the water column using temporary well points is not typically recommended since this drilling method cannot seal off the borehole from other water-bearing zones. As such, use of this method with hollow stem auger equipment is recommended for sampling the uppermost groundwater only.

Once the desired water sampling depth is reached, the well point, constructed of 2-inch OD PVC casing and a 5-foot long PVC screen, will be placed inside the augers. The augers are then pulled up approximately 5 feet as a temporary sand pack is placed in the annulus. The well point will then be purged of at least one borehole volume of water, or until the casing is entirely evacuated, whichever occurs first. Groundwater samples will then be collected as per the methodology described in Section 4.3.

Temporary well points are not proposed as part of the Phase I RI. If proposed for use, temporary well points will be collected at pre-determined locations in accordance with a DEQ approved Work Plan addendum.

4.2 Screening-Level Groundwater Samples from Push Probe Borings

Groundwater samples will be collected from push probe borings by attaching a 4-foot section of stainless steel slotted well screen to the probe and setting the screened interval across or beneath the anticipated groundwater level. Once the desired depth is reached, the outer protective sheath is pulled back to expose the screen. Immediately following well screen placement, a water level measurement will be made with an electric water level meter. Groundwater will be purged from the screen by inserting a disposable polyethylene tube equipped with a check valve down the interior of the probe and a vacuum pump will be used to remove at least 1 borehole volume of water. Samples will be collected via the vacuum pump. Samples for volatile compounds will be collected with polyethylene bailer tubing.

4.3 Groundwater Samples from Monitoring Wells

At least 72 hours following development of the monitoring wells, the groundwater in monitoring wells will be sampled. Prior to sampling, at least three well casing volumes of water will be purged from each well using a peristaltic purge pump equipped with new polyethylene tubing. If water levels fall below 20 feet during purging of any of the wells,

then a decontaminated submersible pump may be necessary for purging and sampling activities. The pH, temperature, and conductivity of the purged water will be measured to assess for stabilization of these parameters. Stabilization is considered to have been met when the last three measured values for each of the above parameters are within 10 percent of each other.

A representative sample of the groundwater will then be obtained using the peristaltic or submersible pump at a low flow rate. For volatile analyses, the groundwater sample will be collected using a new polyethylene bailer. The water will be carefully transferred to appropriate containers. The sampling containers will be completely filled such that no headspace is present that would allow the loss of volatiles. The sample bottles will then be transferred to a chilled container for shipment to the analytical laboratory.

4.4 Water Level Monitoring

Prior to every monitoring well sampling event, and at any other designated water level monitoring events, the static water levels in monitoring wells will be measured to the nearest 1/100th of a foot with a Solinst water level indicator (conductive probe). The water levels will be measured from the north side of the top of the casing where a notch will be cut. The water level from the staff gauge in the Willamette River will also be measured during each water level monitoring event.

5. DECONTAMINATION PROCEDURES

All drilling and push probe equipment will be steam-cleaned between drilling locations to prevent cross-contamination. All soil sampling equipment will be decontaminated prior to each sample by using a detergent (Alconox) solution wash, followed by two separate potable water rinses.

Re-usable push probe water sampling equipment will be decontaminated prior to collecting each sample by using a detergent (Alconox) solution wash, followed by two separate potable water rinses. The peristaltic pump will not need decontamination since water never comes in contact with any re-usable part. In the case where a submersible pump is used, it will be decontaminated as indicated above. If NAPL is encountered during groundwater sampling, then reusable groundwater sampling equipment will be decontaminated with a detergent solution wash, a dilute isopropanol rinse, a two separate potable water rinses.

6. INVESTIGATIVE DERIVED WASTE (IDW)

Investigative derived waste (IDW) will be managed in a manner that is consistent with the U.S. Environmental Protection Agency (EPA) *Guide to Management of Investigation Derived-Wastes* dated January 1992.

All soil cuttings, purge water, decontamination water, and development water generated during the investigation activities, will be containerized and left on-site pending a determination as to appropriate disposition. Disposal of all IDW will take place as soon as practicable following characterization of the waste.

All disposable personal protective equipment (gloves, etc.) and disposable sampling equipment (sleeves, bailers, tubing, etc.) generated during any sampling event will be disposed of at a permitted municipal solid waste disposal facility.

7. QUALITY ASSURANCE PLAN

7.1 Data Quality Objectives

Data gathered during the RI will provide the basis for decisions relating to RI investigation requirements, risk analysis, and remedial measures, if necessary. Specifically, data collected during the RI will be used for a number of purposes:

1) to identify the presence of constituents in known/suspected source areas
2) to determine the nature and extent of contamination
3) to characterize site hydrogeology
4) to identify hot spots
5) to compare to risk-based screening levels
6) to support a risk assessment, if necessary
7) to support a Feasibility Study (FS), if necessary.

The overall data quality objective for the RI is to collect sufficient data of an adequate quality to meet data quality objectives 1 through 6 above. The data quality assurance objectives for this project are to develop and implement procedures to collect representative samples, to provide chemical and physical data of known quality, and obtain chemical data with reasonably achievable method detection limits that are appropriate for risk screening. In order to meet these objectives, all field activities will be conducted according to the methods described in this SAP.

7.2 Quality Control Samples

Three types of QC field samples are proposed for this investigation including field duplicates, trip blanks, and field equipment blanks. Analytical parameters for QC samples may change with each area investigated at the site. The proposed QC samples and analytical parameters for the Phase IA activities are included in Table B1.

One duplicate soil sample will be collected and analyzed for every 20 investigative samples analyzed. One duplicate groundwater sample will be collected and analyzed for every 10 screening-level groundwater samples analyzed and one duplicate groundwater sample will be collected and analyzed for each monitoring well sampling event.

One trip blank sample will be analyzed for every two days of screening-level groundwater sampling and one per monitoring well sampling event. The trip blanks will be prepared by the analytical laboratory, and placed in the sample cooler prior to arrival on-site.

A field equipment blank will be collected for every two days of screening-level groundwater sampling and one per monitoring well sampling event. Following decontamination of the pump, de-ionized water will be passed through the unit, and the effluent water will be collected in appropriate sampling containers.

Laboratory QC for this project will involve standard EPA QC guidelines as described in *Test Methods for Evaluating Solid Wastes - Physical/Chemical Methods* (SW-846) and therefore will not be repeated here. Laboratory QC will include calibration standards, laboratory control samples, reagent blanks, matrix spikes, matrix spike duplicates, surrogate spikes, and laboratory duplicates. Attachment C-1 in Appendix C contains the Quality Assurance Plan and Standard Operating Procedures for Columbia Analytical Services, the chemical analytical subcontractor.

7.3 Sample Handling Procedures

7.3.1 Sample Containers

The sample container, preservation, and holding time requirements for each sample matrix and the anticipated analytical methods are summarized on Table B2. All samples will be transferred to the appropriate sampling containers and placed into a chilled (4°C) transport container for shipment to the on-site mobile laboratory or off-site fixed laboratory. The chilled transport containers (coolers) will be utilized for temporary storage of the samples.

7.3.2 Sample Labels

A sample label will be attached to each sampling container prior to the sampling event. Information to be included on the label will include the following:

1) Sample number
2) Date and time of sample collection
3) Initials of person collecting the sample
4) HAI project number
5) Type of preservative, if any
6) Analyses to be performed

Individual samples will be identified using a unique sample number that includes a sample prefix consisting of the HAI project number and a six digit date code. The prefix will be followed by an individual sample collection number that will be assigned sequentially as the samples are collected (e.g. 4800-000411-001).

7.3.3 Chain-of-Custody Record and Shipment

Appropriate sample Chain-of-Custody forms will be placed in the sample coolers in a plastic bag. An example of the Chain-of-Custody form is attached at the end of this appendix. The coolers will then be sealed with Chain-of-Custody tape for delivery to the laboratory.

7.4 Documentation Procedures

Documentation of field procedures, observations, and measurements will be provided through the use of field logs, chain-of-custody, and photographs.

All data collection activities will be documented using waterproof field forms and indelible black ink. All field entries will be signed, dated, and as detailed and descriptive as possible. If an incorrect entry is made on any form, it will be lined out, the correct information entered, and the correction will be initialed and dated by the person making the correction.

Overall documentation of the nature and timing of field activities will be provided daily with an HAI Project Field Notes form. All borings and wells will be documented with a field Boring Log form. Development, purging, and sampling of any wells or temporary well points will be documented with a Groundwater Sampling Summary form. Investigative wastes will be documented using an IDW Inventory form. Examples of these forms are included as Attachment A.

7.5 Equipment Calibration and Maintenance Procedures

All field equipment will be calibrated prior to use according to the manufacturer's instructions. Users manuals and calibration records will accompany all monitoring equipment into the field and will be stored in the carrying case. Calibrated equipment will be identified by means of an HAI identification number or a manufacturer's serial number. The results of calibrations and records of repair will be maintained in a logbook. Equipment that fails calibration or fails to operate properly will be removed from service, tagged to indicate its condition, and segregated from the operational equipment. Such equipment will be repaired and re-calibrated if possible, or replaced. Preventive maintenance of field equipment is performed according to the procedures indicated in the manufacturer's manuals.

Laboratory analytical equipment and instruments will be calibrated in accordance with the laboratory's internal quality assurance/quality control (QA/QC) program.

7.6 Data Reduction and Validation

As discussed, all field data will be summarized and recorded on appropriate field forms. Descriptive data including soil types, field screening results, observations, and water levels will be summarized in a final appropriate format on boring logs or tables. A review of field QA/QC will be conducted.

Laboratory data will be recorded by a computer system that collects and compiles raw data. The analytical laboratory will conduct necessary QC calculations that will be summarized in final laboratory reports as noted in Section 7.3 All final laboratory reports will be included as an appendix or appendices to the RI report. In addition, all analytical data will be summarized in tabular form.

Analytical data will be assessed to ensure that they are of acceptable quality. The analytical results of samples collected by HAI will be evaluated during the data validation process. The evaluation will include analysis of field and laboratory blanks and duplicates results, surrogate and matrix spike recoveries, detection and quantitation limits achieved, holding times, and equipment calibrations. If the data are not considered to be of adequate quality, an appropriate data qualifier will be assigned. After the data have been reviewed and qualified, the data will be entered into spreadsheets and presented in tabular and graphical formats for interpretation.

7.7 Quality Assurance Audits and Corrective Action

A field QA audit will be conducted during each phase of the RI and will include field sampling, associated sample handling and decontamination techniques to assure that they are suitable to meet project data quality objectives and are consistent with this SAP. Audits will be performed based on a review of field logs, forms, notes, and field inspections. In the event that a field audit indicates work activities are not being performed in accordance with appropriate procedures detailed in this SAP, they will be remedied immediately and the activities evaluated for impact to project data quality objectives. The findings of all field audit procedures will be incorporated into the RI report.

The analytical laboratory QA project manager will be responsible for monitoring consistency within the laboratory QA program. Following receipt, the analytical data and QC data will be reviewed and subject to a data validation and evaluation as discussed in Section 7.7. In the event that a QA problem is identified, the laboratory QA project manager will be notified. Following notification, it is expected that the laboratory will remedy the QA problem; however, in severe cases the laboratory may be dismissed from further services. The laboratory QC results will be included in each analytical report and included in the RI report.

GLOSSARY OF ABBREVIATIONS

ASTM	American Society of Testing and Materials
bgs	below existing ground surface
BTEX	benzene, toluene, ethylbenzene, and xylene
C	degrees centigrade
DEQ	Oregon Department of Environmental Quality
EPA	U.S. Environmental Protection Agency
eV	electron volt
FS	Feasibility Study
ID	inside diameter
IDW	investigative-derived waste
NAPL	non-aqueous phase liquid
OAR	Oregon Administrative Rule
OD	outside diameter
ORS	Oregon Revised Statutes
OWRD	Oregon Water Resources Division
PAHs	polynuclear aromatic hydrocarbons
PCBs	polychlorinated biphenyls
PID	photoionization detector
ppm	parts per million
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
RI	Remedial Investigation
SCP	Site Characterization Plan
SAP	Sampling and Analysis Plan
SPT	standard penetration test
TPH	total petroleum hydrocarbons
USCS	Unified Soil Classification System
UV	ultraviolet
VOCs	volatile organic compounds

TABLE B-2
Sample Container, Preservation, and Holding Time Requirements

Remedial Investigation Work Plan
Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

Project No. 4800

Method Number	Techniqu	Sample Matrix	Sample Container	Preservation	Holding Time
NW TPH Hydrocarbon Identification (HCID)	GC/FID	Soil	8-ounce glass jar	Cool to 4 C	extract within 14 days
		Water	1-liter glass amber bottle	Cool to 4 C	extract within 7 days
NW TPH-G Gasoline Quantification	GC/FID	Soil	8-ounce glass jar	Cool to 4 C	14 days
		Water	(3) 40-ml VOA vials	Cool to 4 C	14 days
NW TPH-Dx Diesel-range and Oil-range Quantification	GC/FID	Soil	8-ounce glass jar	Cool to 4 C	extract within 14 days
		Water	1-liter glass amber bottle	Cool to 4 C	extract within 7 days
EPA Method 8021B Benzene, Toluene, Ethylbenzene, Xylene (BTEX)	GC/PID	Soil	6-inch PVC or brass sleeve capped with teflon tape and plastic end caps	Cool to 4 C	14 days
		Water	(3) 40-ml VOA vials	Cool to 4 C HCl pH<2	14 days
EPA Method 8260B Volatile Organic Compounds (VOCs)	GC/MS	Soil	6-inch PVC or brass sleeve capped with teflon tape and plastic end caps	Cool to 4 C	14 days
		Water	(3) 40-ml VOA vials	Cool to 4 C HCl pH<2	14 days
EPA Method 8270C Semivolitile Organic Compounds (SVOCs) **EPA 8270 SIM** Polynuclear Aromatic Hydrocarbons (PAHs)	GC/MS	Soil	8-ounce glass jar	Cool to 4 C	14 days
		Water	1-liter glass amber bottle	Cool to 4 C	extract within 7 days
EPA Method 8082 Polychlorinated Biphenyls (PCBs)	GC/ECD	Soil	8-ounce glass jar	Cool to 4 C	extract within 14 days
		Water	1-liter amber glass	Cool to 4 C	extract within 7 days
Total Metals: Arsenic, cadmium, chromium, copper, nickel, lead, tin, zinc EPA Method 6010/200.7	ICP	Soil	8-ounce glass jar	Cool to 4 C	6 months
		Water	1-liter plastic	Cool to 4 C HNO3	6 months
EPA Method 7060 - Arsenic					
EPA Method 7421 - Lead					
EPA Method 7470A Mercury	Cold Vapor Analyzer	Soil	8-ounce glass jar	Cool to 4 C	23 days
EPA Method 245.1 Mercury	Cold Vapor Analyzer	Water	1-liter plastic	Cool to 4 C HNO3	23 days

NOTE C = degrees centigrade
ECD = electron capture detector
EPA = U.S. Environmental Protection Agency
FID = flame ionization detector
GC= gas chromatography
HCl = hydrochloric acid
HNO3 = nitric acid
ICP = inductively coupled plasma
PVC = polyvinyl chloride
SIM = Selective Ion Mode
TPH = total petroleum hydrocarbons
VOA = volatile organic analyses

ATTACHMENT B-1
Example Field Logs and Forms

HAHN AND ASSOCIATES, INC.
PROJECT FIELD NOTES

Page ____ of ____

Date : _____
Project: Portland Ship Yard
Project No.: 4800
Project Manager: _____

CONTINUATION SHEET

Time	Task Description

(5/92)

Signed _____

HAHN AND ASSOCIATES, INC.
PROJECT FIELD NOTES

Date : _____
Project: Portland Ship Yard
Project No.: 4800
Project Manager: _____

WORK FORCE	EQUIPMENT	SITE
HAI	Rental/Vendor	Conditions
Subcontractor	Rental/Vendor	Temperature
Subcontractor		

HAI EQUIPMENT

Air Pump (Sensidyne)	Field Office	Measuring Wheel	Pump, Purge	Wtr Filter Unit
Aquifer Test	Gas Meter (GasTech)	OVA (MicroTip/PID)	Pump, Submersible	
Bailer (SS or Teflon)	Generator	Packer	Troll 4000	
Cond/pH/Temp Meter	Hand Auger	Power Tool	Turbidity Meter	
Data Logger (Hermit)	Interface Probe	Pressure Transducer	UV Box	
DO Meter	Magnetometer	Pump, Peristaltic	Water Level Meter	

HAI SUPPLIES

Bailer (disposable)	Distilled Water (gal)	Locks	Tubing (well)
Bentonite (bags)	Gloves, disposable (pr.	Resp. Cartridges (pr.)	Tyvek, coated
Booties (pr.)	Gloves, Edmont (pr.)	Sample Tubes	Tyvek, uncoated
Booties (pr.)	Gloves, Solvex (pr.)	Sensidyne Tubes	Water Filters
Concrete (bags)	Locking Caps	Stakes	

ACTIVITIES

Leave Office:	Arrive Site:	Leave Site:	Arrive Office:	
Respirator Use/ Hours:___	Minutes:___	Odometer Out:	Odometer In:	Total Mileage:

Time	Task Description

(5/98)

Signed _____

HAHN AND ASSOCIATES, INC.

Environmental Management

434 NW Sixth Avenue, Suite 203 · Portland OR 97209

(503) 796-0717 · Fax (503) 227-2209

Laboratory

Lab Project No.

CHAIN OF CUSTODY

Chain of Custody No.

Project Manager		Liquid with Sediment Sample	Samples Received at 4C (Y or N)
Project No.	4800	Test Filtrate Test Sediment Test Both	Appropriate Containers Used (Y or N)
Project Name	Portland Ship Yard, Portland, Oregon	Multi-Phase Sample	Provide Verbal Results (Y or N) No
Collected by		Test One (which) Test Separately Shake	Provide Preliminary Fax Results Yes

Comments	Matrix				Analyses to be Performed									RUSH	Remarks
	Soil	Water	Other	Number of Containers											
Lab ID / **Sample #** / **Date** / **Time** / **Sample Description**															

Relinquished by	Company	Date	Time	Received by	Company
Relinquished by	Company	Date	Time	Received by	Company
Relinquished by	Company	Date	Time	Received by	Company

HAHN & ASSOCIATES, INC. 434 NW Sixth Avenue Portland, Oregon (503) 796-0717	BORING NUMBER: _____		
	HAI LOGGER:	DRILL START	DRILL FINISH
PROJECT: Portland Ship Yard 5555 North Channel Avenue Portland, Oregon	SAMPLING METHOD: DRILLING METHOD: EQUIPMENT TYPE DRILLER:	Time: Date:	Time: Date:
PROJECT #: 4800	DRILLING CONTRACTOR:		

ABANDONMENT DETAILS	SAMPLE NUMBER	TIME	HEADSPACE (ppm)	LAB RESULTS TPH (ppm)	SPT (blows/0.5 feet)	RECOVERY (%)	DEPTH (feet)	GROUNDWATER	STRATA (USCS)	BORING DIAMETER: CASING DIAMETER: SURFACE ELEVATION: TOP OF CASING ELEVATION: SOIL DESCRIPTION
							1			
							2			
							3			
							4			
							5			
							6			
							7			
							8			
							9			
							10			
							11			
							12			
							13			
							14			
							15			
							16			
							17			
							18			
							19			
							20			

HAHN & ASSOCIATES, INC. 434 NW Sixth Avenue Portland, Oregon (503) 796-0717	BORING NUMBER: _____		
	HAI LOGGER:	DRILL START	DRILL FINISH
PROJECT: Portland Ship Yard 5555 North Channel Avenue Portland, Oregon	SAMPLING METHOD: DRILLING METHOD: EQUIPMENT TYPE DRILLER:	Time: Date:	Time: Date:
PROJECT #: 4800	DRILLING CONTRACTOR:		

ABANDONMENT DETAILS	SAMPLE NUMBER	TIME	HEADSPACE (ppm)	LAB RESULTS TPH (ppm)	SPT (blows/0.5 feet)	RECOVERY (%)	DEPTH (feet)	GROUNDWATER	STRATA (USCS)	SOIL DESCRIPTION
							21			
							22			
							23			
							24			
							25			
							26			
							27			
							28			
							29			
							30			
							31			
							32			
							33			
							34			
							35			
							36			
							37			
							38			
							39			
							40			

BORING DIAMETER:
CASING DIAMETER:
SURFACE ELEVATION:
TOP OF CASING ELEVATION:

HAHN & ASSOCIATES, INC. 434 NW Sixth Avenue Portland, Oregon (503) 796-0717	MONITORING WELL NUMBER OWRD #	MW - 1s	
PROJECT: Porland Ship Yard 5555 North Channel Avenue Portland, Oregon PROJECT: 4800	HAI LOGGER: Joan Doe SAMPLING METHOD: 2 & 3-inch Split Spoon Sampler DRILLING METHOD: Hollow Stem Auger EQUIPMENT TYPE Mobile B-59 DRILLER: John Doe DRILLING CONTRACTOR: Good Drilling Company	DRILL START Time: 11:00 Date: 14-Feb-99	DRILL FINISH Time: 15:00 Date: 14-Feb-99

BORING DIAMETER:	10 1/4-inch OD
CASING DIAMETER:	2-inch ID
SURFACE ELEVATION:	346.25 feet MSL
TOP OF CASING ELEVATION:	345.92 feet MSL

WELL CONSTRUCTION DETAILS	SAMPLE NUMBER*	TIME	HEADSPACE (ppm)	FLUORESCENCE	SPT (blows/0.5 feet)	RECOVERY (%)	DEPTH (feet)	GROUNDWATER	STRATA (USCS)	SOIL DESCRIPTION
Colorado 10/20 Sand Pack	041	1215			45	100	21			Sandy GRAVEL with cobbles and trace silt - as above, no odor, no discoloration, no sheen.
2-inch ID 10 slot PVC Screen			19	No	>50	20	22			
							23		GW	
	042	1230			28	100	24			Sandy GRAVEL with cobbles and trace silt - as above, no odor, no discoloration, no sheen.
			16	No	30	20				
Cap							25			
							26			
							30			
							31			
							32			
							33			Materials:
							34			1 flush monument 1 lock 1 10-foot PVC casing 1 5-foot PVC casing 1 10-foot 0.010-slot PVC screen 12 50# bags 10/20 colorado silica sand 9 50# bags bentonite chips 2 50# bags cement
							35			
							36			
							37			
							38			
							39			* Sample Number Prefix: 3484-990214-
							40			

EXAMPLE

HAHN & ASSOCIATES, INC.	MONITORING WELL NUMBER	MW - 1s	
434 NW Sixth Avenue	OWRD #		
Portland, Oregon	HAI LOGGER: Joan Doe	DRILL START	DRILL FINISH
(503) 796-0717	SAMPLING METHOD: 2 & 3-inch Split Spoon Sampler		
PROJECT:	DRILLING METHOD: Hollow Stem Auger	Time:	Time:
Porland Ship Yard	EQUIPMENT TYPE: Mobile B-59	11:00	15:00
5555 North Channel Avenue	DRILLER: John Doe	Date:	Date:
Portland, Oregon	DRILLING CONTRACTOR: Good Drilling Company	14-Feb-99	14-Feb-99
PROJECT: 4800			

BORING DIAMETER:	10 1/4-inch OD
CASING DIAMETER:	2-inch ID
SURFACE ELEVATION:	346.25 feet MSL
TOP OF CASING ELEVATION:	345.92 feet MSL

WELL CONSTRUCTION DETAILS	SAMPLE NUMBER*	TIME	HEADSPACE (ppm)	FLUORESCENCE	SPT (blows/0.5 feet)	RECOVERY (%)	DEPTH (feet)	GROUNDWATER	STRATA (USCS)	SOIL DESCRIPTION
Flush Monument										Ground surface
Concrete							1		GP	Asphalt GRAVEL - brown, moist, dense, non-plastic, poorly graded, (fill).
							2			SILT - brown, moist, firm, slightly-plastic, no odor, no discoloration.
							3		ML	
							4			
							5	2/21/97	GW	Sandy GRAVEL with some silt - brown, moist, very dense, non-plastic, moderately graded, no odor, no discoloration, no sheen.
					5	100				
3/4-inch Bentonite chips	036	1115			20	100	6			
			45	No	38	100				
2-inch ID Blank PVC Casing							7			
					6	100	8			
	037	1130			27	100				Sandy GRAVEL with some silt - as above, wet, no odor, no discoloration, no sheen.
			22	No	50	20	9			
							10	2/14/97		
					4	100			GW	Sandy GRAVEL with some silt - as above, no odor, no discoloration., no sheen.
	038	1140			5	100	11			
			41	No	28	10				
	039	1150								
							15			
					15	100				Sandy GRAVEL with cobbles and trace silt - brown, wet, very dense, non-plastic, moderately graded, no odor, no discoloration, no sheen.
Colorado 10/20 Sand Pack	040	1200			18	100	16			
			32	No	12	20				
2-inch ID 10 slot PVC Screen							17			
	-	1210	-	-	>50	-	18		GW	
							19			
							20			

EXAMPLE

INVESTIGATIVE DERIVED WASTE (IDW) INVENTORY FORM

Project Name Portland Ship Yard
Project Locatio 5555 N Channel Avenue
Portland, Oregon

Project Number 4800
Project Manager
Date

	<— Fill Out In Field				Follow-Up —>				
Container #	Container Type	Contents	Date of Generation	Labeled As	Waste Determination	Date of Disposal	Disposal Method	Disposal Location	Comments
CHOICES	55-gal drum 32-gal drum Other (explain)	Soil Cuttings Purge Water Decon Water PPE Sampling Supplies Other (explain)		Hazardous Special Non-hazardous Other (explain)	Hazardous Special Non-hazardous Other (explain)		Landfill Treatment On-site Disposal	Hillsboro TPS Arlington Other (indicate)	

Updated By: Date: Updated By: Date:

Updated By: Date: Updated By: Date:

Well Development Summary Sheet

WELL NUMBER _____

General Information	
Project Name	Portland Ship Yard
HAI Project No.	4800
Date	
Developing Personnel	
Purge Method	

Purge Volume Calculation

Total Well Depth (ft)	Static Water Level (ft)	Water Column (ft)	Convert Factor (gal/foot)	One Well Vol (gal)	TEN Well Vol (gal)

2' well = 0.17 gallons/linear ft 4" well = 0.66 gallons/linear ft

Well Purge Data **Total Volume to Purge =**

Time	Volume Purged (gallons)	Conductivity (uS/cm) x100 / x1000	Temperature degrees (C / F)	pH	Comments

Purge Water Disposition

Drum No.	Storage Tank No.	On Ground	Other

Monitoring Well Sampling Summary Sheet

WELL NUMBER _____

General Information	
Project Name	Portland Ship Yard
HAI Project No.	4800
Date	
Sampling Personnel	
Purge Method	
Sampling Method	

Sample Information
Sample Date
Sample Time
Sample Number

Purge Volume Calculation

Total Well Depth (ft)	Static Water Level (ft)	Water Column (ft)	Convert Factor (gal/foot)	One Well Vol (gal)	Three Well Vol (gal)

2' well = 0.17 gallons/linear ft 4" well = 0.66 gallons/linear ft

Sample Containers

Number	Type	Preservative	Analytical Parameters

Well Purge Data **Total Volume to Purge =**

Time	Volume Purged (gallons)	Conductivity (uS/cm) x100 / x1000	Temperature degrees (C / F)	pH	Comments

Purge Water Disposition			
Drum No.	Storage Tank No.	On Ground	Other

Temporary Well Point
Sampling Summary Sheet

WELL POINT NUMBER _____

General Information	
Project Name	Portland Ship Yard
HAI Project No.	4800
Date	
Sampling Personnel	
Purge Method	
Sampling Method	

Sample Information
Sample Date
Sample Time
Sample Number

Purge Volume Calculation

Total Well Depth (ft)	Static Water Level (ft)	Water Column (ft)	Convert Factor (gal/foot)	One Well Vol (gal)	Three Well Vol (gal)

2' well = 0.17 gallons/linear ft 4" well = 0.66 gallons/linear ft

Sample Containers

Number	Type	Preservative	Analytical Parameters

Well Purge Data **Total Volume to Purge =**

Time	Volume Purged (gallons)	Conductivity (uS/cm) x100 / x1000	Temperature degrees (C / F)	pH	Comments

Purge Water Disposition

Drum No.	Storage Tank No.	On Ground	Other

Table B-1
Proposed Phase Ia Laboratory Analysis Summary
Remedial Investigation Work Plan
Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

HAI Project No. 4800

Area of Investigation	Investigative Method	Number Soil Borings	Sample Matrix	Analytical Parameters	Tentative Number of Soil Samples to be Analyzed				
					Investigative Samples	Field QA/QC Samples			Matrix Total
						Duplicate	Trip Blank	Equip. Blank	
Ballast Water Treatment Plant (BWTP)	Push Probe	12	Soil	Metals	24	2			26
				TPH	24	2			26
				BTEX	12	1			13
				PAHs	12	1			13
				PCBs	12	1			13
		12	Groundwater	Metals	12	1		1	14
				BTEX	12	1	1	1	15
				PAHs	12	1		1	14
N. Channel Avenue Fabrication Site	Push Probe	15	Soil	Metals	18	2			20
				TPH-Dx	18	2			20
				PAHs	9	1			10
				PCBs	9	1			10
		3	Groundwater	Metals	3	1		1	5
				PAHs	3	1		1	5
				VOCs	3	1	1	1	6
Building 73	Push Probe	3	Soil	Metals	3				3
				TPH	3				3
				PAHs	2				2
				VOCs	3				3
		3	Groundwater	Metals	3				3
				PAHs	3				3
				VOCs	3				3
Building 4	Push Probe	5	Soil	Metals	5				5
				TPH	5				5
				PAHs	2				2
				VOCs	5				5
		5	Groundwater	Metals	5				5
				PAHs	5				5
				VOCs	5				5
Paint Shed/Blast Booth Area	Push Probe	5	Soil	Metals	5	1			6
				TPH	5	1			6
				PAHs	2	1			3
				VOCs	2	1			3
		5	Groundwater	Metals	5	1		1	7
				PAHs	5	1		1	7
				VOCs	5	1	1	1	8

bgs = below ground surface
BTEX = benzene, toluene, ethylbenzene, and xylene
PAHs = polynuclear aromatic hydrocarbons
PCBs = polychlorinated biphenyls
QA/QC = quality assurance / quality control
TPH = total petroleum hydrocarbons by hydrocarbon identification (HCID) method
TPH-Dx = diesel and oil-range total petroleum hydrocarbons
VOCs = volatile organic compounds

Table B-1
Proposed Phase Ia Laboratory Analysis Summary
Remedial Investigation Work Plan
Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

HAI Project No. 4800

Area of Investigation	Investigative Method	Number Soil Borings	Sample Matrix	Analytical Parameters	Tentative Number of Soil Samples to be Analyzed				
					Investigative Samples	Field QA/QC Samples			Matrix Total
						Duplicate	Trip Blank	Equip. Blank	
Building 43, 50, 80 Area (Steam Cleaning Basin)	Push Probe	5	Soil	Metals	6	1			7
				TPH-Dx	6	1			7
				PAHs	3	1			4
				VOCs	6	1			7
		2	Groundwater	Metals	2				2
				PAHs	2				2
				VOCs	2				2
Electrical Substations	Hand Auger	36	Soil	TPH	36	2			38
				PCBs	36	2			38
Old Boiler Area	Push Probe	1	Soil	Metals	2				2
				TPH	2				2
				PAHs	1				1
		1	Groundwater	Metals	1				1
				PAHs	1				1
				VOCs	1				1
Former Hazardous Waste Storage Area	Push Probe	4	Soil	Metals	4	1			5
				TPH	4	1			5
				PAHs	2	1			3
				PCBs	2	1			3
				VOCs	4	1			5
		4	Groundwater	Metals	4	1		1	6
				PAHs	4	1		1	6
				VOCs	4	1	1	1	7

bgs = below ground surface
BTEX = benzene, toluene, ethylbenzene, and xylene
PAHs = polynuclear aromatic hydrocarbons
PCBs = polychlorinated biphenyls
QA/QC = quality assurance / quality control
TPH = total petroleum hydrocarbons by hydrocarbon identification (HCID) method
TPH-Dx = diesel and oil-range total petroleum hydrocarbons
VOCs = volatile organic compounds

APPENDIX C – SURFACE WATER AND SEDIMENTS SAMPLING AND ANALYSIS PLAN

APPENDIX C:

PORTLAND SHIPYARD REMEDIAL INVESTIGATION COMBINED SAMPLING AND ANALYSIS PLAN AND QUALITY ASSURANCE PROJECT PLAN FOR SURFACE WATER AND SEDIMENTS

DRAFT, JANUARY 15, 2000

Prepared For:

Port of Portland
PO Box 3529
Portland, OR 97208

and

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222 Kenyon St NW
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ACRONYMS

ABN	Acid/Base/Neutral compounds
AETs	Apparent Effects Threshold
ASTM	American Society for Testing Materials
AVS/SEM	Acid Volatile Sulfides/Simultaneously Extracted Metals
BFB	
CAS	Columbia Analytical Services
COC	Chain-of-Custody
COI	Contaminant of Interest
CLP	Contract Laboratory Program
CORPS	U.S Army Corps of Engineers
CRM	Certified Reference Material
DEQ	Oregon Department of Environmental Quality
DFTPP	
DGPS/GPS	Differential Global Positioning System
DMR	Dinnel Marine Research
DQO	Data Quality Objective
EPA	U.S. Environmental Protection Agency
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/FID	Gas Chromatography/Flame Ionization Detector
GC/FPD	Gas Chromatography/Flame Photometric Detector
GC/MS	Gas Chromatography/Mass Spectrography
GPC	Gel Permeation Chromatography
HCl	Hydrochloric Acid
ICP	Inductively Coupled Argon Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectroscopy
mL	Milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSS	Marine Sampling Services
NAD	North American Datum
NAS	Northwestern Aquatic Sciences
NOAA	National Oceanic and Atmospheric Administration
NWTPH-HCID	Northwest Total Petroleum Hydrocarbons-Hydrocarbon Identification
OD	Outside Diameter
ODEQ	Oregon Department of Environmental Quality
PCBs	Polychlorinated Biphenyls
PPM	Parts Per Million
PRQL	Practical Quantitation Limits
PSAMP	Puget Sound Ambient Monitoring Program
PSDDA	Puget Sound Dredged Disposal Analysis

PSEP	Puget Sound Estuary Program
PSY	Portland Shipyard
QA1	Quality Assurance Level "1" (as specified by WDOE)
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QLs	Quantitation Limits
RPD	Relative Percent Difference
SIM	Select Ion Monitoring
SPI	Sediment Profile Imaging
SRM	Standard Reference Material
SOP	Standard Operating Procedure
SOW	Statement of Work
SPI	Sediment Profile Image
TBT	Tributyltin
VOA	Volatiles Organics Analysis
WDOE	Washington Department of Ecology

1. FIELD SAMPLING PLAN

The Portland Shipyard (PSY) Remedial Investigation (RI) is designed to meet the objectives described in the Site Characterization Plan section of the Remedial Investigation/Feasibility Study Work Plan for the Portland Shipyard (PSY). This Sampling and Analysis Plan (SAP) was developed in response to the State of Oregon Department of Environmental Quality (DEQ) File Review Memorandum regarding the Swan Island Portland Shipyard (File Memo-ECSI No. 271). DEQ reviewed the Port of Portland's PSY Sediment Investigation Report (SEA 1998) and determined that the Port had completed a substantive portion of the RI. However, the File Review Memorandum identified several sediment data gaps that needed to be filled to complete the RI. Striplin Environmental Associates (SEA) will be responsible for implementing this SAP.

The field sampling plan discusses the additional locations, analyses and samples needed to fill two data gaps identified in DEQ's File Review Memorandum. The first data gap is the collection and analysis of additional bulk samples of surface and subsurface sediment to complete the identification of the aerial and vertical extent of chemicals of interest. This data gap is discussed in Section 1.1. The second data gap is the determination of the bioaccumulation potential of the sediment. This is discussed in Section 1.1.3 and in Section 4.

This document describes methods for the collection, analysis and quality control of samples obtained in and around the shipyard. These samples will be used to complete the characterization of the aerial and vertical extent of contaminated sediments near the facility and the bioaccumulation potential of the sediment.

1.1 SAMPLING LOCATIONS AND RATIONALE

1.1.1 Selection of Surface Station Locations

Eighteen surface sediment samples will be collected at specific locations in and around PSY to fill data gaps identified in the DEQ file memorandum (Figure C-1, Table C-1).

Five areas are designated for sampling to complete aerial extent analyses:

1. Swan Island Lagoon between Berths 304 and 308

   Two additional surface sediment stations will be located in this area. The first will be between historical Stations PSY10 and PSY 14. The second will be between PSY 3 and PSY 10. These stations are being established

to fill data gaps along the PSY berths. Large tankers and others vessels were blocking access to these berths at the time of the PSY Sediment Investigation in March and April 1998.

2. Berth 302 to further define the PCB area at PSY 14

   Four additional stations around at and Station PSY 14 (one at PSY14, two along side and one offshore) to define the extent of PCB contamination.

3. Dry dock basins

   Six stations will be located in the three dry dock basins. Two stations will be positioned under each of Dry Docks 1, 3 and 4 because sampling under the dry docks has not occurred since the early 1990's. A seventh station will be established off the mouth of Dry Dock 4 to determine the lateral distribution of COIs near Dry Dock 4.

4. Willamette River side of the Shipyard

   Four stations will be established at and around historical Station DM-H. Station DM-H was located offshore of the Ballast Water Treatment Plant (BWTP) outfall and there was some indication that moderately high levels of COIs were found at this station.

5. Swan Island Lagoon at Berth 311

   One station will be established at the location of Berth 311. This station will be established between Stations PSY-5 (Berth 311) and PSY-1 to further define the distribution of COIs in surface sediments near Berth 311 and the City of Portland storm water outfall located adjacent to PSY-1.

1.1.2 Selection of Subsurface Station Locations

Nine subsurface stations will be established and co-located with the surface sample stations to further determine the vertical distribution of COIs in the sediment (Figure C-2, Table C-2).

Cores will be taken to the maximum depth possible, generally the depth where refusal of the core occurs. Each core will be cut into four-foot sections for compositing and analysis to assess the vertical distribution of chemicals of concern.

1. Swan Island Lagoon at Berth 311

   A single subsurface station will be established at the location of Berth 311. This subsurface station will be co-located with the surface station described above. This station will be established between Stations PSY-5 (Berth 311) and PSY-1 to further define the distribution of COIs in

subsurface sediments near Berth 311 and the City of Portland storm water outfall located adjacent to PSY-1.

2. Berth 302 to further define the PCB area at PSY 14

   Two additional subsurface stations will be established to determine the vertical distribution of PCBs near PSY 14. One will be established at the location of station PSY 14 and the second will be located offshore of PSY 14.

3. Dry dock basins

   Four subsurface stations will be established to determine the vertical distribution of chemicals of interest (COI) in or near the dry dock basins. One station will be established under Dry Docks 1, 3 and 4. Coring will be done using a drilling rig to sample through access hatches on the floor of each dry dock. The forth-subsurface station will be established in the area west of the location of PSY 36 at the mouth of Dry Dock 4.

4. Small Boat Basin Offshore of the BWTP

   Two subsurface stations will be established offshore of the BWTP to further define the subsurface distribution of COIs in this area.

1.1.3 Selection of Stations for Bioaccumulation Testing

A phased approach will be used to select surface sediment stations for bioaccumulation testing. The first phase will involve the collection and analysis of surface sediment samples for bulk chemistry. From among these stations, several will be selected for bioaccumulation testing. The selected stations will include stations that span a range of COI concentrations. The second phase will be the collection of fresh sediment at the stations selected for bioaccumulation testing. The selection of stations and COIs for testing will occur after DEQ has finalized its procedures and methods for evaluating the risks posed by bioaccumulative contaminants. The proposed approach to bioaccumulation testing will be described in the Phase II RI Work Plan addendum that will be submitted to DEQ for review and approval.

1.2 SAMPLE DESIGNATIONS

Each sample designation reflects a short prefix identifying the sampling event and station number, and is followed by a letter describing the sampling horizon [S for surface (0-10 cm), A for 0-4 ft interval, B for 4-8 ft, and C for 8-12 ft]. Because this is the second round of sampling at the PSY, the survey name will be PSY(2). For example, sample PSY(2)-01S is decoded as PSY(2) = Portland Shipyard-second round, 01 = Station No.

01, and S= surface, and for a core sample, PSY(2)-01A is taken from the 0-4 ft interval, PSY01B is taken from the 4-8 ft interval. Following this scheme the station numbers will take over where the previous station numbering stopped.

1.3 STATION POSITIONING

A differential global positioning system (DGPS) will be used for precision navigation. DGPS consists of a GPS receiver on the sampling platform and a differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver located on the sampling vessel. The GPS receiver routes latitude and longitude to an integrated navigation system, which displays the vessel's position in plain view. Navigation data such as range and bearing from the target sampling location are provided at a user-defined scale to guide the vessel's pilot to the desired location.

Differential GPS can provide accuracies on the order of ±1-5 meters. Positioning accuracies on the order of ±1-3 meters will be achieved by avoiding periods when the signal is weak. The GPS system will not be used when it is not receiving strong satellite signals (i.e., when the vessel is up against a large dry dock). When this occurs, distances to the station will be measured from known landmarks. Actual geodetic coordinates (NAD 1927) for each surface and subsurface station are provided in Tables C-1 and C-2, respectively.

1.4 SAMPLING VESSEL

The *R/V Nancy Anne* will be used to collect all surface grab and subsurface core samples. This vessel is a 36-foot shallow draft catamaran with a 2,000-pound capacity winch. It is powered by twin 120 hp Volvo stern drives. The *R/V Nancy Anne* is owned and operated by Bill Jaworksi of Marine Sampling Services (MSS), Gig Harbor, Washington.

1.5 SURFACE SEDIMENT SAMPLING

Surface Sample Collection. Collection of undisturbed sediment requires that the sampler:

- Create a minimal bow wake when descending.
- Form a leak proof seal when the sediment sample is taken.
- Prevent winnowing and excessive sample disturbance when ascending.

- Allow easy access to the sample surface.

Surface (0 - 10 cm) sediment samples will be collected using a 0.3 m^2 hydraulically powered grab sampler provided by Marine Sampling Systems. The power grab sampler is designed to consistently collect undisturbed samples to the required depth below the sediment surface. The grab sampler is fitted with screened top doors covered with rubber flaps. Upon descent, the flaps are forced open to minimize the bow wake and, upon ascent, the flaps are forced closed to prevent sample winnowing.

The power grab will be lowered by a hydraulic crane and winch at a controlled speed of approximately 4 ft/sec until near the bottom, at which time the lowering speed was reduced to a speed of approximately 1 ft/sec. The power grab is opened and closed electronically by switches controlled by the captain.

A position fix will be taken each time the sampler hits bottom at a station. After impact, the grab will be retrieved and gently positioned on a processing table. If necessary, the boat will be maneuvered to minimize vessel rolling to prevent the grab from swinging. After being brought aboard and secured on the table, the sampler will be visually evaluated for the absence of water leaking from the sides or bottom due to rocks, sticks or organisms caught in the jaws of the grab.

Grab Sample Acceptability. After recovery, the grab will be evaluated for sample acceptability. The hinged lids on top of the sampler will be opened and the sample will be inspected for the following criteria (PSEP 1986):

- Sediment does not extrude from the upper surface of the sampler and is not pressed against the top of the sampler.
- Sediment surface is relatively flat and undisturbed (not sloped with partial sample or washed near the sides of the grab).
- No water leakage from the sampler is allowed. Overlying water is present to indicate minimal leakage.
- Overlying water is not excessively turbid.
- The penetration depth is at least 5 inches (12 cm).

If a sample fails to meet all of these criteria, it is rejected. If an acceptable sample is not obtained after a reasonable number of attempts, the station position is moved, following approval of the Field Manager.

Once the grab sample is deemed acceptable, penetration depth is measured and recorded on the surface sample log sheets (Figure C-3). Sample characteristics, such as color,

odor, sediment type, and presence of fauna or flora, will also be noted on the field log sheets. Overlying water will be slowly siphoned off from near the edges of the sampler.

Grab Sample Processing. Sediment grab samples are processed according to the following step-by-step procedure:

1. The samples are first collected for volatile organics analysis (VOA) and sulfides. The jars will be filled so that there was no headspace. The sulfide sample will be preserved with zinc acetate
2. The top 10-cm of sediment will be collected using a clean stainless steel spoon or spatula. Sediment in contact with the edges or bottom of the grab will not be collected.
3. Sediment is transferred to a clean stainless steel bowl and covered with aluminum foil.
4. The grab sampler will be redeployed until enough sediment is obtained for the required analyses.
5. The composite sample will be stirred until the sample is of uniform color and texture. If materials (e.g., shells, rocks) are removed it will be noted on the field log.
6. The jars for the remaining chemical, and bioaccumulation analyses are filled.
7. Each glass container is sealed in a plastic bag in case of breakage. The samples will be packed in an ice chest to minimize the chances of breaking.
8. Excess sediment from the grab and composite samples will be returned to the collection site.
9. The grab sampler and equipment are then decontaminated to prepare for sampling at the next station.

1.5.1 Diver Collected Samples from Under the Dry Docks

Divers will sample surface sediments located within the PSY dry dock basins. Samples will be collected using a hand-held stainless steel corer having a 4-inch diameter. A valve at the top of the corer will be opened to allow water to escape and prevent compression of the sediment surface. The corer will be inserted in the sediment to a depth of 10-cm using a gentle twisting motion. Once the corer is completely pressed into the sediment, the diver closes the valve, slides the bottom retention plate into position to contain the sediment, and slowly extracts the corer from the sediment. The diver will return the sealed corers to the sampling vessel where it is transferred to a processing crew.

A diver core sample will be considered acceptable if the following conditions are met:

- The corer penetrated to the minimum acceptable sampling depth
- Minimal water is present within the sample core
- No sample is lost prior to compositing.

If the sample is acceptable, care will be taken to remove all overlying water from the corer and the sediment will be transferred from the corer into a stainless steel bowl. The physical characteristics of the sediment are recorded on the field log sheets, and sediments will be homogenized using the same methods described for surface grab samples. Adequate volumes of sediment will be collected to perform the analyses for the target chemicals listed in Table C-3.

1.6 SUBSURFACE SEDIMENT SAMPLING

Core Collection. The vibracorer will be provided by Mr. Bill Jaworski of MSS. The MSS custom-built vibracorer deployed from the *R/V Nancy Anne* can obtain sediment cores up to 20 ft long. The vibracorer uses a hydraulic system that vibrates and drives a length of 4-inch outside diameter (OD) aluminum tubing into the sediment. A continuous sediment sample is retained within the tubing with the aid of a stainless steel core cutter/catcher.

Sediment recovery measurements will be used to cut sections of the core to desired lengths, usually 4-foot long sections. The *in situ* depth to the top of the section will be recorded for each section on the core log sheet (Figure C-4). Before the tube is cut, a label identifying the station and core section will be securely attached to the outside of the casing at the top of each section, and wrapped with transparent tape to prevent loss or damage of the label. The core-sections will be labeled with the boring number and the top and bottom depths below the mudline that the sample was collected from.

Sediment at the end of each tube section will be visually classified for qualitative sample characteristics. Changes from the top to the bottom of each section of the tube will be noted and recorded on the core log sheet. Empty tubing will be removed to assure that each section is full of sediment. The core ends will then be covered with aluminum foil, a protective cap and duct tape to prevent leakage. The core sections will be stored upright in a container chilled with ice or "blue ice" to approximately 4°C. The cores will be transported to the onshore sample processing facility twice daily.

Subsurface Sampling Beneath Dry Docks. Sampling of subsurface sediment beneath the dry docks will be accomplished using a portable or truck-mounted drill rig. The drilling truck will be craned onto the dry dock, secured, and drilling will occur through openings within the dry dock.

A hollow-stemmed auger will be used for sampling. Sampling within the hollow-stemmed auger will be accomplished with either a Shelby tube or a split spoon sampler.

For both types of sampling implements, the hollow-stem auger will be advanced to the level within the sediment column at which the intended sample starts (e.g. if the 2-4 ft sediment interval below mudline is to be collected, the auger would be advanced to –2 ft below the sediment surface). Once the auger is at the desired level within the sediment column, the sampling device within the auger will be advanced into the sediment. By stopping the auger at the upper limit of the intended sample interval, the shelby-tube or split-spoon sampler will then be advanced beyond the auger and into undisturbed sediment.

Shelby tubes are typically hydraulically advanced into the sediment column beyond the auger tip. Shelby tubes typically collect a 2-ft sample interval and are 3 inches in diameter. The shelby tube sample offers a more undisturbed sample and greater sample volumes than a split-spoon sampler. It is most effective in unconsolidated or poorly consolidated sediments.

A split-spoon sampler is more effective at collecting consolidated and stiff subsurface sediments than a shelby-tube type sampler. If used, the split spoon will be advanced into the sediment by hammering the drill rod with a known weight. The number of blows needed to advance the split spoon to the desired sampling interval will be recorded in the log and will provide a relative measure of the shear strength and stiffness of the native sediments. Split-spoon samplers are generally 20-24 inches in length and 2.5 inches in diameter.

Core Sample Acceptability. As the core is brought aboard it will be visually evaluated for penetration and loss of sediment from the tube. Overlying water should be present but should not be excessively turbid. Caution will be used to prevent disturbance of the surface of the sediment when it is placed in a horizontal position during removal from the coring tripod. The core catcher will be inspected for rocks or other obstacles that may have plugged the core while penetrating. A core will be rejected if there is doubt about its representativeness.

Sediment depth in the core is used to determine if the desired depth has been sampled. If recovery is poor, collection of another core may have to be attempted at that station. The longer core of the two will be chosen as the primary core to be analyzed. The second core is retained in case problems, such as excessive disturbance, is discovered during core processing. Secondary cores will not used if the first core is acceptable.

Core Compaction. The vibracore is equipped with a transducer to monitor drive length (core penetration). Compaction will be estimated (instrument recovery/drive length) and

used to determine *in situ* sampling depths. Recovery will be measured by dividing the core percent length (measured using a ruler) by the drive length.

Core Sample Processing. Core sections will be extruded in the laboratory by attaching the core tube at an angle to a vibrating table and then tapping the tube with a mallet. This process produces a generally intact core for visual classification of the sediments with depth and subsequent collection of sediment for chemical analysis. Step by step core processing procedures are outlined below:

1. Secure the core to a vibrating table. Remove the duct tape and aluminum foil from the top of the tube, being careful not to disturb or lose sediment.
2. Extrude core sections by tilting and/or vibrating the core section and letting the sediment slide out slowly. If necessary, a clean, fabricated stainless steel plunger will be used to push the core onto the table.
3. Exclude the sediment in contact with the edges of the core tube by peeling or scraping off a layer of sediment, approximately 0.5 cm in depth, around the exposed circumference of the sample. Slice 2.5 cm of sediment off each end of the section that may have been in contact with the pipe cutter and plunger.
4. Note stratification of color and texture on the core log sheet. Note debris, odor, sheen, biological features, sediment density and other distinguishing characteristics.
5. Transfer the sediment to a stainless steel bowl, excluding the portions that may have been in contact with the core tube. Stir with a stainless steel spoon until the sample is of uniform color and texture. Remove debris (e.g., rocks, shells) and note their removal on the core log.
6. Collect samples for chemical analyses (leaving headspace) and place them in a refrigerator.
7. Collect sample for grain size analysis. If necessary, scrapings from the sides may be used if they are representative of the sample.
8. Seal each glass jar in a plastic bag and place sample in ice chest or refrigerator. Pack the samples to minimize the chances of breaking. Decontaminate the processing equipment prior to processing the next core.

Note that samples will be composited over a 4-ft length of the core. Samples will not be composited over more than one 4-ft core section, nor will sediments from different core stations be composited.

1.7 FIELD QUALITY CONTROL SAMPLES

Field quality control samples help determine if other sources of variability, outside of laboratory sample handling and manipulations, are present. For this study, blind field replicates and field sample splits will be collected. One station will be selected for collection of these samples prior to sampling.

Field replicate samples are used to measure and document the repeatability of field sampling methods and techniques. Field replicates represent sediment samples collected and composited independently of the primary sample using the same sampling techniques as the primary sample. Field replicates will be collected immediately following the primary sample, at the same location as the primary sample. The field replicate sample will be analyzed for all the analytes that the original sample was analyzed for. Field replicates will be used to determine field variability (including the homogeneity of sample locations and sample processing techniques) and will also be used to provide insight to laboratory variability.

Blind field split samples will also be collected from the replicate stations. Field split samples will be collected from the same homogenate as the original sample. These samples will provide insight to sample handling and processing techniques and analytical variability.

1.8 EQUIPMENT DECONTAMINATION

Contamination of samples must be avoided during sample collection and processing activities. All sampling equipment, sample containers, utensils, instruments, working surfaces and other items that may come in contact with the sediment will be made of a noncontaminating material (e.g., glass, stainless steel, teflon). Decontamination of stainless steel bowls, utensils, and the core catcher will be performed before sampling at each station and between the processing of each composite core sample.

Standard decontamination procedures will be as follows:

- Rinse with tap water or water provided by the sampling vessel
- Wash with brush and phosphate free detergent, rinse with distilled water
- Rinse with 0.1N nitric acid and then with distilled water
- Rinse with methanol (this step will be omitted for equipment used to collect sediment for analysis of volatile organic compounds).

Clean sample handling equipment will be wrapped in aluminum foil, with the dull side facing the equipment. Field personnel will wear disposable gloves that are rinsed with

distilled water before and after handling each sample as appropriate, to help minimize sample contamination.

The power grab sampler will be decontaminated between sampling stations by scrubbing it with a brush and a phosphate free detergent to remove excess sediment, followed by a rinse with site water. As an added measure, when sediment is removed from the grab sampler, sediment that is in contact with the sides of the sampler will not be collected.

During coring, new core tubes will be used for each deployment. The core tubes will be decontaminated before sampling by scrubbing them with a brush and detergent and then rinsing. Sediment in contact with the sides of the tube and at the ends of the sections will not be retained. The pipe cutter will be kept clean with a cloth and occasionally cleaned with water. Extrusion equipment (e.g., plungers and trays) will be scrubbed with a brush and water and lined with clean aluminum foil when possible.

1.9 SAMPLE CONTAINERS AND STORAGE

Storage requirements and container volumes and types are described in Table 7-1 of Appendix D of the Columbia Analytical Services (CAS) QAPP (see Attachment C-1). In all cases, the amount of sediment collected will be more than that required for analysis and quality control purposes. Additionally, sediment will be archived in case analyses need to be repeated or if additional analyses are requested at a later date.

All containers will be filled leaving some headspace. Samples will be stored either in refrigerators or on ice in coolers until delivered to the laboratory. CAS personnel will pick up the chemistry samples at the end of each sampling day. Bioaccumulation samples will be kept refrigerated (4°C) at the field staging area until delivery or pickup is made to the respective biological laboratory. Once at the chemical or biological laboratory, samples will be refrigerated at 4°C until used for the chemical and biological tests.

1.10 SAMPLE HANDLING AND TRANSPORT

On completion of a final inventory of the samples, each jar will be placed into a plastic bag and sealed. Samples will be immediately placed on ice. A chain-of-custody form will be completed and provided to CAS and NAS when the samples are received at the end of each day.

Samples for analysis will be picked up each afternoon by CAS and NAS personnel and transported immediately to their laboratory.

For samples that will be stored at the staging area, signed custody seals will be used to seal each sample container to ensure that sample integrity is not compromised. Sample refrigerators will also be maintained in a locked and restricted building at the PSY.

1.11 CHAIN-OF-CUSTODY PROCEDURES

Chain-of-custody (COC) procedures will be followed from the time of sample collection to the conclusion of laboratory analysis.

Field COC procedures include:

- Label sample containers with preprinted labels with station and sample information plus the analytical parameter(s) that the container contents are intended for. Date, time and sampler information will be written on the label in the field.
- Complete chain-of-custody forms for all samples en route to processing facility, laboratory, or storage. Upon transferring samples to the laboratory sample custodian, the cruise leader or designated staff sign, date and note the time of transfer on the chain-of-custody form.
- Ship samples in ice chests sealed with custody seals, unless relinquished directly to a laboratory representative. The integrity of the seals is established at the laboratory by the laboratory sample custodian.
- Ensure that the samples are in possession or view of field staff or secure storage at all times.
- Transport samples to the laboratory as soon as possible, observing appropriate preservation and holding-time requirements.
- Transfer custody of the samples to the appropriate COC lockers and refrigerators. Document the transfer on the appropriate COC record-keeping form and/or logbook.
- Notify the appropriate people that the samples had arrived.

Upon receipt of the samples at the laboratory, the laboratory sample custodian will inventory the samples by comparing sample labels to those on the COC document. The custodian will enter the sample number into a laboratory tracking system by project code and sample designation. The custodian will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing samples in an appropriate secure area.

1.12 FIELD LOG BOOK AND SAMPLE DOCUMENTATION

A field log book will be used to record observations and pertinent information at each sampling station. It will be a bound document containing individual field and sample log forms. Information included in the log book will include personnel, date, time, station position, station designations, sampler, types of samples collected, and general observations.

Additional information will be entered on surface sample description forms or core log sheets (Figures C-3 and C-4). The following information will be included:

- General site and/or project number
- Sample number
- Collection date
- Collection time
- Water depth
- Tide height
- Sediment depth
- Penetration depth (grab sampler) or sample recovery (core)
- Personnel collecting samples
- Sediment type (e.g., particle size fractions, debris in the sample, sample color and odors)

1.13 SAMPLING AND ANALYSIS SCHEDULE

Sampling will take approximately seven days to complete. Surface grab sampling will occur prior to subsurface coring. The grab sampling schedule will be restricted to a maximum of 8 samples/day because the analytical laboratory (CAS) can handle only 8 pore water tributyltin samples/day due to limitations of equipment used for the pore water extraction.

2. LABORATORY ANALYSIS AND QUALITY CONTROL

In accordance with the objectives laid out for this project, the analytical plan and associated quality assurance and quality control (QA/QC) procedures comply with the analytical protocols in the Environmental Protection Agency's Contract Laboratory Program (CLP) (EPA 1993) and the Puget Sound Estuary Program (PSEP) guidance (EPA 1989a,b). Puget Sound Dredge Disposal Analysis (PSDDA) guidance (Corps of Engineers, 1989 1998) is also satisfied by these requirements.

The following sections describe the laboratory analysis and QA procedures for analytical chemistry.

2.1 ANALYTICAL CHEMISTRY METHODS AND PROCEDURES

Columbia Analytical Services (CAS) of Kelso, Washington, will perform metals, semivolatiles, volatiles, PCB Aroclors, pesticides, sulfides, total solids, grain size and total volatile solids and total organic carbon analyses on sediment samples. CAS will also perform pore water extraction with subsequent analysis for ammonia and tributyltin.

Chemical analyses of sediment and pore water are required to meet the objectives of this project. Laboratory quality assurance will be implemented and maintained as described in this plan and according to the laboratory's Quality Assurance (QA) program and standard operating procedures (SOPs).

For sediments, the analytical methods, quality control (QC) measurements and criteria are based on a combination of current CLP and Methods for Solid Waste Analysis (SW-846), PSEP guidance, and PSDDA requirements. Laboratory deliverables are consistent with the requirements outlined by the PSDDA program, known as a QA1 deliverable and those in Appendix G of the Portland Harbor Sediment Management Plan (PHSMP) (DEQ 1999). Some of the analytical methods cited below for pore water are fundamentally SW-846 and National Oceanic and Atmospheric Administration (NOAA) methods, but have been revised to meet data quality objectives (DQOs) for this project. The modifications are consistent with the PSEP guidelines and achieve PHSMP requirements.

Target analytes and their respective quantitation limits (QLs) required for this project, and analytical methods, are listed in Tables C-3 and C-4 respectively. The required QLs were based on the 1999 PHSMP and the PSDDA Chemical Guidelines for Puget Sound and Grays Harbor/Willapa Bay. The quantitation limits established for pore water analyses are low enough to support the further development of site specific sediment quality criteria for COIs . While best efforts will be made to achieve the project DQOs,

there may be cases in which efforts may not meet the specified goals. Any limitations in data quality due to analytical problems (e.g., due to highly contaminated samples) will be clearly identified in the data validation report.

Modifications to established analytical methods for some analytes may be necessary to achieve project QLs. In some cases, the sample size and final volume of the digestate or extract may have to be adjusted to achieve these QLs. For example, for analysis of cadmium, arsenic, and silver in sediments by ICP/MS, it may be necessary to increase the final digestate volume from 100 milliliters to 500 milliliters to reduce interference from the sample matrix. This modification is a common standard operating procedure (SOP) in many environmental analytical laboratories to improve detection limits and low-level precision, and is accepted by the CLP.

It is anticipated that QLs for all analytes in a sample may not be attained due to chemical interferences, especially in those samples exhibiting elevated levels of other target analytes.

Some detail is provided in the following section for those analytical methods requiring modification of standard (or otherwise referenced) procedures. Detailed and complete SOPs, laboratory QA program plans, and example deliverables for CAS are provided in the attachment to this SAP.

2.1.1 Methods for Analysis of Pore Water Samples

Pore Water Extraction. Pore water extracted from sediment will be analyzed for tributyltin (TBT) and ammonia. At least four of the appropriate centrifuge tubes will be loaded with homogenized sediment from the same station, sealed, and centrifuged. The number of tubes centrifuged for a given station will depend on the pore water yield. Large amounts of sediment are needed to collect the 400 to 500 mL required for tributyltin and ammonia analyses.

The polycarbonate tubes will be centrifuged at 2475 *g* for 30 minutes. The supernatants will then decanted to a clean polycarbonate tube and centrifuged for an additional 30 minutes. The resulting supernatant will be transferred to a clean polycarbonate container for analysis. The pore water for tributyltin analysis will be acidified with 1 milliliter of concentrated HCl per 100 mL of sample.

A small amount of pore water will be transferred to a pre-cleaned plastic bottle for ammonia analysis after preservation with 0.5 mL of 9N sulfuric acid. Samples will be stored in a cool, dark location.

Tributyltin. Tributyltin analyses will be performed on pore water using the SW-846 methods, modified according to Krone (1989). The modifications include use of Grignard reagent for derivatization followed by GC/Flame Photometric Detector (GC/FPD). Quantitation limits will be in the range 0.025-0.05 ug/l.

Ammonia. Ammonia in pore water will be analyzed as nitrogen by ion-specific electrode. A 50-ml sample will be modified using 1 ml 10 N sodium hydroxide. After mixing the sample well, a previously calibrated ammonia electrode will be inserted into the sample. Ammonia as nitrogen will be reported to a limit of 0.3 mg/L (ppm). This method is based on EPA Method 350.3.

2.1.2 Methods for Analysis of Sediment Samples

Grain Size. Grain size analysis will be accomplished on all project samples according to PSDDA guidelines using ASTM D-422-63 (wet sieve with hydrometer) with modifications described in the PSEP protocols and guidelines (PSEP 1986). Eight class fractions will be determined. Peroxide oxidation will not employed so that biological aggregates are not broken apart. Results will be expressed by class percentage (reportable to 0.01 percent) in the following fractions: gravel (6-1 ø), very coarse sand (1-0 ø), coarse sand (0-1 ø), medium sand (1-2 ø), fine sand (2-3 ø), very fine sand (3-4 ø), sand (1-4 ø), silt (4-8 ø), clay (>8 ø), and fines (>4 ø). Results will be presented in tabular format and, for selected samples, plotted on semilogarithmic paper as percent fines by weight versus grain size.

Total Organic Carbon. Total organic carbon content will be measured according to guidelines found in the PSEP protocols (PSEP 1986b) and options recommended in Michelsen (1992). Sample pretreatment with HCl will be required to liberate inorganic carbon (principally carbonates). TOC analyses will be performed by oxidizing the sample at ~850° C and then measuring CO_2 via infrared spectrophotometry. Results will be expressed in terms of carbon per dry weight of the unacidified sample.

Metals. Metals will be determined according to a modification of EPA Method 200.8 (ICP/MS). Sample digestion will be accomplished with HNO_3/H_2O_2 and final digestate volumes may have to be increased to 500 milliliters to reduce matrix and inter-element interferences. Initial and continuing calibrations, mass calibration, resolution check and stability checks will be conducted according to EPA Method 200.8. A selected list of elements will be chosen based on historic data and the requirement for rapid turnaround time from the laboratory. Results will be reported as ug/kg dry weight.

Acid, Base, and Neutral Extractable Organic Compounds. Selected acid, base, and neutral (ABN) extractable organic compounds in sediments will be analyzed by a

modification of the methodology found in SW-846 methods 3550/8270B. Procedural modifications included the following:

- Extract 30 - 50 gram (wet wt) samples via sonication/homogenization followed by gravity settling and separation.
- Dry primary extract over anhydrous Na_2SO_4.
- Use gel permeation chromatography (GPC) for cleanup and removal of elemental sulfur (S_x).
- Adjust final extract volumes to give sufficient sensitivity and instrumental response without overloading.
- Establish GC/MS initial calibration with a minimum of calibration points in the range of 2-100 ug/ml for all target analytes.
- Conduct continuing calibration for all target analytes and surrogate compounds.

Both matrix spike and surrogate spike compounds will be added prior to sample extraction, as required by the analytical method.

Chlorinated Pesticides and PCBs. Chlorinated pesticides and PCBs (as Aroclors) will be analyzed by methodology specified in the most recent SW-846 Methods 3550/8081 and 3550/8082, with the following procedural enhancements:

- Extract approximately 30 grams of sample via sonication/homogenization.
- Remove elemental sulfur (S_x) from the sample extract during GPC cleanup. Additional S_x removal may be required using chemical agents, at the discretion of the analyst.
- Conduct florisil column chromatography of extracts for pesticide analysis (as required based on chromatographic interferences).
- Adjust final extract volumes to achieve analyte PRQLs and to prevent instrumental overloading.

Some samples will be analyzed for PCBs only and will be subjected to sulfuric acid cleanup to remove potential interferences and ensure the accurate identification and quantitation of PCBs. All associated QC will be as required by SW-846 methods. Data deliverables will be as required by the PHSMP data reporting requirements.

Volatile Organic Compounds. Volatile organic compounds will be analyzed in sediments using the purge and trap GC/MS technique. The analytical method is as described in SW-846 method 8260. All quality control criteria will be similar to that of the CLP SOW with exceptions as provided in the CAS SOP VOC-8260A.

Tributyltin. Sediment samples from surface and core composites will be analyzed for bulk sediment tributyltin using SW-846 methods, modified according to Krone (1989). The modifications include use of Grignard reagent for derivatization followed by GC/Flame Photometric Detector (GC/FPD). Quantitation limits in the range of 10 - 25 ug/kg will be necessary. The analytical SOP is found in the attachment to this SAP. Deliverables will be consistent with the PHSMP data requirements.

2.1.3 Chemical Analysis of Tissue Samples

Columbia Analytical Services (CAS) will conduct laboratory testing to determine selected COI concentrations in organisms following bioaccumulation testing. Following completion of bioaccumulation testing exposure, frozen tissue samples from reference and test samples will be shipped directly to CAS by the biological laboratory. At CAS, tissue samples will be homogenized prior to analysis to assure representative sub-sampling for selected COIs and total lipids measurements.

Tissue samples will be analyzed for TBT using solvent extraction, derivatization, and GC/FID. Samples are dried with muffled, anhydrous sodium sulfate. Following the addition of surrogate compounds (trypropyltin chloride and tripentyltin chloride), tissues are extracted with methylene chloride that contains 0.1% (wt./vol.) tropolone. After solvent exchange into hexane, extracts are derivatized with hexylmagnesium bromide in ether via a Grignard reaction. The Grignard reagent is synthesized by CAS because commercially available reagents are not of acceptable purity. Tissue extracts are cleaned by elution through Florisil® columns. The extracts are analyzed by GC/FPD or GC/MS analysis.

Samples will be analyzed using EPA Method 1668 - Isotope dilution and high resolution gas chromatograph/ high resolution mass spectrometry (HRGC/HRMS). Congeners quantified using HRGC/HRMS include IUPAC # 77, 126, 169, 105, 114, 118, 123, 156, 157, 167, 170, 180, and 189. In addition, congener #81 that is not included in Method 1668, will be quantified using an appropriate internal standard. Bulk reference material 1944 (NY/NJ Waterway sediment) will be purchased from the National Institute of Standards and Technology (NIST) and analyzed as a QC measure.

2.2 QUALITY CONTROL REQUIREMENTS AND INTERNAL QUALITY CONTROL CHECKS

Quality control procedures for laboratory analysis will be consistent with the requirements described in the CAS SOPs, which are presented as part of the laboratory's QA program plan in Attachment C-1 to this SAP. Methods for establishing the quality of laboratory measurements and sample results generally conformed to CLP quality control requirements or other quality criteria as described by the QA plan. Some modifications

will be made to expand the range of instrumental calibrations, reduce quantitation limits, and establish precision at quantitation levels below those of the CLP.

Data validation and reporting of data quality will conform to the criteria of the EPA Data Validation Functional Guidelines for metals and organics (EPA 1988a,b) as can be determined by the evaluation of standard report formats without raw analytical data.

All QC measurements and data assessment for this project will be conducted on samples from and within batches of samples from this project alone; samples from other projects will not be mixed with samples from this project for assessment of data quality. PSEP guidelines will be used for evaluating and establishing data quality for analytes/parameters that are not addressed by the EPA CLP guidance documents.

Sample Handling and Storage. Procedures for laboratory sample handling and storage are documented in a written CAS SOP attached to this SAP. Table 7-1 of that document summarizes the requirements for sample containers, preservation, and holding times. One addition to Table 7-1 is the 24-hour holding time for extraction of pore water from sediments when tributyltin analysis is performed. This holding time was established based on the desire to minimize exposure of pore water to possible tributyltin sources (such as paint chips) as soon as possible after sampling to better reflect *in situ* conditions.

Instrument Calibration and Checks. Instrument calibration and checks will conform to analytical protocol requirements and laboratory analytical SOPs, found in the CAS QA Plan.

Methods for Assessing Precision. Precision will be assessed by examining analytical and field variability using the following three types of measurements: laboratory splits, field sample splits and field replicates. Method, holding and field rinsate blanks will also be analyzed to ensure sampling precision.

Laboratory Sample Splits

To assess analytical variability, analyses of laboratory-generated sample splits will be performed. Laboratory splits will be performed once per batch of 20 samples, and relative percent differences (RPDs) will be calculated. Variabilities in organic compound analyses will be evaluated by analysis of matrix spike (MS) and matrix spike duplicate (MSD) samples. Samples for inorganic analyses will be split in the laboratory and separately analyzed without matrix spikes. Conventional parameters will be analyzed in triplicate. Quality control objectives and limits for analysis of laboratory splits will be consistent with CLP requirements.

Field Sample Splits

Verification of laboratory measurements of sample analytical variability will be accomplished for sediments by analyzing blind field samples that will be generated in the field by subsampling the composited sample. These samples will help determine if other sources of variability outside of laboratory sample handling and manipulation are present or unaccounted for. Field sample splits will be generated at one station.

Field Replicates

An assessment of variability associated with combined analytical and site environmental variabilities will be accomplished by analysis of field replicates. Station replicates will be collected and composited independent of the primary sample and associated sample splits, yet will be collected from the same station. Extra sample volume will be collected at two locations identified to the laboratory for use in generation of laboratory splits and MS/MSD according to analysis needs.

This scheme for assessing both analytical and sampling variabilities has proven useful in the Puget Sound Ambient Monitoring Program (PSAMP), some PSDDA programs, during the 1998 PSY Sediment Investigation, and at least one CERCLA site. Results from splits and replicates will be taken into consideration during the assessment of the overall uncertainty and significance of the data used in site characterization.

Method, Holding, and Field Blanks

Introduction of contaminants during sampling and analytical activities will be assessed by the analysis of blanks. Laboratory method blanks, generated by the laboratory, will be analyzed at a minimum frequency of one per analytical batch of 20 for all chemical parameter groups. An additional "holding" blank will be generated and analyzed for the single batch of volatile organic compound analyses (VOAs) only. These blanks will be used to determine if volatile chemicals were introduced to samples during holding or storage prior to analysis. A field blank, consisting of sampling equipment rinsate water will be generated for all chemical parameter groups upon completion of sampling. Data will be qualified during validation if contaminants are found in any of these blanks.

Methods for Assessing Accuracy. Accuracy will be assessed in terms of analytical recovery for all chemical analytes by the use of independent reference material or matrix spikes. Analyte recovery from the reference material or the MS will be measured at a minimum frequency of one per batch of up to 20 samples. Data will be qualified during data validation based upon recoveries from these analyses. Quality control objectives will be based on CLP criteria.

CAS-generated acceptance criteria and requirements will be employed for the analyses conducted in this program and will be used to support the evaluation of laboratory results (Table C-4) during data validation. Table C-4 also presents the QC criteria for evaluating surrogate recoveries during low-level pore water and organic compound analyses. CLP requires qualification of results when surrogate recoveries fall outside of acceptance limits.

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted in accordance with the QC requirements identified in the CAS laboratory QA Plan. In addition, each of the specified analytical methods provides protocols for proper instrument calibration, setup, and critical operating parameters.

2.2.1 Laboratory Sample Custody Procedures

Upon receipt by the laboratory, each sample will be checked for physical integrity and logged into a laboratory information management system. Samples will be handled and stored so as to maintain sample integrity before and after analysis. Specific SOPs for sample handling, tracking, storage and custody are found in the attachment to this SAP.

2.2.2 Laboratory Data Deliverables

Laboratory data deliverables will consist of hardcopy documentation of the laboratory procedures used and sample results, and will be consistent with PHSMP data requirements. For contaminants not normally analyzed using CLP protocols, documentation will be prepared according to the deliverables described in the attachment to this SAP. Documentation will be sufficient to allow a CLP-style of review during data validation. This information will be sufficient to review the data with respect to the following:

- All GC/MS raw data files will be provided to SEA on electronic media (CD Rom if possible)
- Holding times and conditions
- Instrument calibration (including standard reference material)
- Detection/quantitation limits
- Surrogate recoveries
- Laboratory split analyses (duplicates and MS/MSDs)
- Precision and accuracy
- Completeness

- Data report formats.

Electronically formatted data (diskette or e-mail) deliverables will be used to expedite data review and validation.

2.2.3 Data Reduction, Validation, and Reporting

Data reduction, evaluation, and reporting performed at the laboratory will generally be in conformance with the CLP statements of work for organic and inorganic analyses, or will be based on the laboratory SOPs when CLP procedures are not available or specified. As discussed in the preceding section, specific deliverables will be required for reporting PHSMP quality level data.

The laboratory may assign data flags, or qualifiers, following CLP protocols for organic and inorganic analyses. The laboratories will be required to immediately notify the analytical chemistry QA Manager when any QC measurements are consistently outside of project QC criteria or DQOs.

Electronically formatted data deliverables will be generated and delivered to the analytical chemistry QA Manager. Complete manual data validation will be performed on 100 percent of the data by the analytical chemistry QA Manager. Data validation and reporting will be accomplished for all analytical parameters including conventional analytes. Hardcopy data deliverables and documentation will be archived for all laboratory results and procedures, and will be made available to Bridgewater Group, the Port of Portland and to DEQ upon request.

The organics data will be evaluated in general accordance with EPA's *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis* (EPA 1988b). Inorganics data will be validated in general accordance with EPA's *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis* (EPA 1988c). Chemical data from non-CLP procedures will be reviewed with regard to the following, as appropriate to the particular analysis:

- Holding times and conditions
- Conformance with required analytical protocol(s)
- Instrument calibration
- Blanks
- Detection/quantitation limits
- Recoveries of surrogates and/or matrix spikes (MS/MSDs)

- Variability for duplicate analyses
- Completeness
- Data report formats.

In addition to the general reporting requirements identified above, PHSMP data delivery requirements for chemical variables are described below.

Laboratory Data Deliverables for Organic Compounds

The following items will be required from the laboratory for completion of analytical reporting:

- A cover letter referencing the procedure used and discussing any analytical problems, deviations and/or modifications, will be signed by a laboratory representative certifying to the quality and authenticity of data as reported
- Report of sample collection, extraction and analysis dates, including sample holding conditions
- Final extract volumes (and dilutions required), sample size, wet-to-dry weight ratios, and the instrument practical detection/quantitation limit for each analyte
- Analyte concentrations with reporting units identified, including data qualification in conformance with the CLP SOW (including definition of data descriptor codes)
- Quantification of analytes in all blank analyses, as well as identification of the method blank associated with each sample
- Recovery assessments and a replicate sample summary [included all surrogate spike recovery data with spike levels/concentrations for each sample and all MS/MSD results (recoveries and spike amounts)].

The following items will be required to be kept on file by CAS, but will be not required for the data report. Any or all of these items might be required if unexplained errors are detected in the data package.

- Reconstructed ion chromatograms for GC/MS analyses for each sample and standard calibration
- GC/ECD and/or GC/FID chromatograms for each sample and standard calibration
- Raw data quantification reports for each sample and calibrations, including areas and retention times for analytes, surrogates and internal standards
- A calibration data summary reporting calibration range used and a measure of linearity [include DFTPP and BFB spectra and compliance with tuning criteria for GC/MS]

- Report of tentatively identified compounds with comparison of mass spectra to library/reference spectra where applicable.

Laboratory Data Deliverables for Metals

The following items will be required from the laboratory for completion of analytical reporting:

- A cover letter referencing the procedure used and discussing any analytical problems, deviations and/or modifications will be signed by a laboratory representative certifying to the quality and authenticity of data as reported
- Report of sample collection, digestion and analysis dates, with sample holding conditions
- Recovery assessments and a replicate sample summary for each sample, if performed, all matrix spike results (recoveries and spike amounts) and laboratory control sample analytical results].
- Tabulation of instrument and method practical detection/quantitation limits
- Tabulated results for samples in units as specified; including data qualification in conformance with the CLP SOW, including definition of data descriptor codes

The following items are required to be kept on file at CAS, but are not required for the data report. Any or all of these items might be required if unexplained errors are detected in the data package.

- Results of all method QA/QC checks including ICP Interference Check Sample and ICP serial dilution results
- Raw data quantification report for each sample
- A calibration data summary reporting calibration range used and a measure of linearity, where appropriate
- Final digestate volumes (and dilutions required), sample size, and wet-to-dry weight ratios
- Quantification of analytes in all blank analyses, as well as identification of the method blank associated with each sample.

2.2.4 Performance and System Audits

The analytical chemistry QA Manager will oversee the activities of all analytical chemistry support employed in this project. This oversight will be achieved through on-

site inspections and reviews of analytical facilities prior to and during analyses of project samples.

Prior to initiating laboratory analyses, a QA evaluation of the laboratory will be performed in a manner similar to those procedures used for a CLP-type systems audit. The laboratory QAPP and SOPs will be evaluated prior to sample submittal. Continuing performance audits will be conducted on a regular basis to ensure the laboratory is providing data of known and sufficient quality. Independent standard reference materials for tissues and sediment (where available for the analytes of concern) will be used, at a minimum, at the beginning and end of each task or phase of the project as an independent assessment of the analytical process.

The frequency of on-site audits will depend on the type of interaction and communications the analytical chemistry QA Manager experiences with the laboratory staff, and on the frequency of observations of noncompliance with QC criteria and SOPs. The QA Manager's interaction with the laboratory will focus on coordination, management, and assessment of performance, and on the rapid institution of corrective actions, if required.

2.2.5 Preventive Maintenance

Preventive maintenance in the laboratory will be the responsibility of laboratory personnel and analysts. This maintenance includes routine care and cleaning of instruments, and inspection and monitoring of carrier gases, reagents, solvents, reference materials, and glassware used in the analyses. All maintenance of instruments and procedures will be documented in maintenance log/record books. The laboratory has SOPs for preventive maintenance (attached to this SAP).

2.2.6 Assessment of Data Quality

The assessment of data quality will be based on criteria developed to address project DQOs. Laboratory performance and data assessment will consist of on-site audits and data evaluation during the data validation activities as described above. Laboratory data will be qualified with the use of data descriptors assigned by the laboratory and during independent data validation.

Analytical Precision. Qualification of laboratory results due to exceedance of criteria associated with measurements of precision will be accomplished by determining relative percent differences (RPDs) using field and laboratory sample split analyses. The following equation will be used to calculate the RPD:

$$RPD = (C_1-C_2) \times 200\% / (C_1+C_2), \text{ where:}$$

C_1 = larger of the two observed concentrations
C_2 = smaller of the two observed concentrations.

Analytical and environmental variability will be assessed via blind replicate sample results. These data will be used to determine the overall precision and variability associated with the entire analytical and sampling process. It is anticipated that environmental variability will exceed that due to the analytical process alone. Attempts will be made to quantify the amount of environmental and laboratory variabilities.

Analytical Accuracy. Analytical accuracy will be assessed in terms of analyte recoveries determined during spiked sample analyses and with the use of commercially available reference materials (i.e., SRMs and CRMs). For spiked samples the percent recovery (% R) can be used as a direct measure of accuracy. It is calculated as:

$$\%R = X-X^1 / TV \times 100\%$$

X = Concentration of the analyte recovered
X^1 = Concentrations of unspiked anlayte
TV = True value of amount spiked

$$\%R = (S-U) \times 100\% / C_{SA}, \text{ where:}$$

S = Measured concentration in spiked sample
U = Measured concentration in unspiked sample
C_{SA} = Actual concentration of spike added.

Laboratory results will be assessed and qualified in accordance with CLP requirements by the use of surrogate compound recoveries for organic compounds and matrix spike recoveries for inorganic parameters.

Analytical Completeness. Analytical completeness will be assessed as the ratio of acceptable measurements obtained to the total number of planned measurements for an activity. Completeness (C) is defined as:

% C = (No. of data points within target QC limits) x 100% / (Total No. of data points)

2.2.7 Corrective Actions for Unacceptable Data

Continuous data assessment and comparison of data precision, accuracy, and completeness to the data acceptance criteria and project DQOs will be undertaken. The Laboratory QA Coordinator will keep the analytical chemistry QA Manager apprised of the laboratory's QC status during all analytical events. An assessment of the problem and

institution of corrective action will follow any significant or consistent deviation from acceptance criteria and analytical goals. Specific corrective actions are outlined in each respective CLP SOW or laboratory SOP and may include but are not limited to the following:

- Identify the source of the nonconformance
- Reanalyze sample(s) if holding time criteria permit
- Retrieve archived sample(s) for analysis (each sample collected has an associated archived sample for use as sample backup, primarily for extractable organics and/or metals analyses)
- Reanalyze sample(s) following resampling
- Evaluate and/or amend sampling and analytical procedures
- Accept noncompliant data and apply qualifier(s) to indicate level of uncertainty.

2.2.8 Quality Assurance Reports to Management

After data delivery and validation, a review of data quality for each analytical task will be generated by the analytical chemistry QA Manager. This report will summarize the quality of validated data, present results of system and performance audits, and assess data usability for the project.

3. LABORATORY METHODS FOR BIOACCUMULATION TESTS

Bioaccumulation testing will be conducted by Northwest Aquatic Sciences (NAS) in Newport OR. A 28-day bioaccumulation test will be conducted with the oligochaete *Lumbriculus variegatus* (EPA/Corps 1998, EPA 1994) and/or the clam *Corbicula fluminea* (widely used in the investigation of geographical distributions of contaminants in local biota). The tests will determine the bioaccumulation potential of selected COIs by comparing the concentration of these contaminants in the tissue of organisms exposed to test sediments to that observed in organisms exposed to reference sediments. Draft test protocols are presented in Attachment C-2.

3.1 PRE-TEST QUALITY ASSURANCE PROCEDURES

Mr. Peter Striplin of SEA will be the project biological QA Manager. To ensure the production of technically defensible bioaccumulation data, Mr. Striplin will be responsible for instituting a QA/QC program prior to initiation of the tests. This program will include review of the project-specific SOP and QAPP by the contracting laboratory. It will also include pre- and during-test laboratory audits.

The biological QA Manager, prior to commencement of testing will conduct an audit of the laboratory. Each audit will include a tour of the physical facility and review of the laboratory's QA/QC program, SOPs, and project filing system. Interviews will be conducted with laboratory staff. Each audit will be conducted using guidance from EPA's *Manual for the Evaluation of Laboratories Performing Aquatic Tests* (EPA 1990).

In addition to the pre-test audit, unannounced spot audits will be conducted during the performance of the tests. During these spot audits, the laboratory will allow the QA Manager to have complete access to the laboratory and its personnel.

3.2 TEST PROCEDURES

General guidance for conducting freshwater bioaccumulation tests, with specifics on *Lumbriculus variegatus*, is found in *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Testing Manual* (EPA/Corps 1998) and in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates* (EPA 1994).

Tests will be conducted using large aquaria containing test or reference sediment and filled with overlying water. The test organisms will be randomly distributed into test chambers. Reference sediments will contain approximately the same sediment grain size (percent fines) as the test sediment. Organisms exposed to reference sediments will be used for an endpoint comparison of tissue chemistry with organisms exposed to test sediment.

Water quality parameters during testing are similar to those of bioassay testing and include:

- The correct temperature and pH range
- Adequate oxygen levels
- Proper lighting
- The correct hardness range
- The absence of, or insignificant concentrations of, toxicants such as ammonia.

Tissues will be composited by organism for each sediment test at the end of the 28-day exposure period. The criterion for test acceptability is adequate mass of both organisms at test completion for analysis of selected COIs. The composite tissue samples will be frozen and sent by NAS to CAS for chemical analysis.

3.3 DATA REPORTING REQUIREMENTS

The biological laboratory will document all activities associated with the sample analyses and will prepare a written report. As a minimum, the following will be included in the report:

- Results of the laboratory bioaccumulation analyses and QA/QC results. Raw data will be legible or typed.
- All protocols used during analyses, including explanation of any deviation from the approved sampling plan.
- Chain of custody procedures, including explanation of any deviation from the identified protocols.
- Location and availability of data, laboratory notebooks, and chain-of-custody forms.
- Locations of bioaccumulation test organism acquisition, and negative control sediment and water acquisition.
- Reference sample locations and water depth.

3.4 QUALITY ASSURANCE REVIEW

All data submitted by the laboratory will undergo a quality assurance review. At a minimum, the submitted data will be reviewed for the following:

- **Data Completeness.** Defined as the amount of data obtained versus the amount of data originally intended to be collected. For this program, 80 percent will be considered acceptable.
- **Data Quality Objectives.** Data will be reviewed for compliance with the parameters established in the specific test protocols. These may include, but are not limited to the following:
  - Tests conducted within specified holding times
  - Test organism mortalities/abnormalities exceeding performance criteria
  - Out-of-range water quality parameters
  - Lack of randomization
  - Lack of required reference, control, or reference toxicant exposures
  - Reference toxicant results outside of specified ranges.

3.5 CORRECTIVE ACTION FOR UNACCEPTABLE DATA

Tests that do not meet completeness and DQO objectives will either be qualified or be rerun. The conditions under which retesting could be required include:

- Required reference or control sediments not used
- Test organism mortality in control or reference sediments exceeded acceptable limits
- Test organisms not all the same species
- Water quality parameters consistently out of range
- Sediment holding times exceeded.

The conditions under which retesting may be required or data may have to be qualified include:

- Lack of test array randomization or testing that was not blind
- Test chambers not identical, or were broken or misplaced
- Excessive test organism mortality in a single replicate of a control
- Test organisms not randomly assigned to test chambers or not from same population
- Holding times were exceeded
- Brief episodes of out of range water quality parameters
- Test monitoring was not documented or incomplete
- Sediment holding times were exceeded
- Sediment storage conditions were out of acceptable ranges.

4. REFERENCES

Krone, C.A., D.W. Brown, D.G. Burrows, R.G Bogar, S-L Chan, and U. Varansi. 1989. A method for analysis of butyltin species and measurement of butyltin in sediment and english sole livers from Puget Sound. Marine Environmental Research 27: 1-18.

Michelsen, T.C. 1992. Technical Information Memorandum. Organic carbon normalization of sediment data. Washington State Department of Ecology, Olympia, WA.

Northwestern Aquatic Sciences (NAS). 1998. Test Protocol: Freshwater amphipod, *Hyalella azteca*, 10 day sediment toxicity test. NAS Protocol NAS-XXX-HA4b. Prepared by NAS, Newport, OR.

Northwestern Aquatic Sciences (NAS). 1998. Test Protocol: Freshwater midge, *Chironomus tentans*, 10 day sediment toxicity test. NAS Protocol NAS-XXX-CT4b. Prepared by NAS, Newport, OR.

Parametrix (no date). Procedures for conducting sediment toxicity tests using the Saline Extract Microtox (Protocol 1020a). Prepared by Parametrix, Inc. Kirkland, WA.

PTI Environmental Services. 1989. Puget Sound Dredged Analysis Guidance Manual: data quality evaluation for proposed dredged material disposal projects. Final Report. Prepared for the Washington State Department of ecology, Olympia, WA.

Puget Sound Dredged Disposal Analysis (PSDDA). 1989. Management Plan Report-unconfined open-water disposal of dredged material, Phase II (North and South Puget Sound. Puget Sound Dredged Disposal Analysis series. Cooperatively published by the U.S. Army Corps of Engineers, Seattle District, U.S. EPA Region 10, Washington State Department of Ecology and the Washington State Department of Natural Resources.

Puget Sound Dredged Disposal Analysis (PSDDA). 1998. Chemical guidelines for Puget Sound and Grays Harbor/Willapa Bay.

Puget Sound Estuary Protocols (PSEP). 1986. Recommended protocols for measuring selected environmental variables in Puget Sound sediment and tissue samples. Final report. Prepared for the U.S. Environmental Protection Agency, Region 10, Office of Puget Sound, Seattle, WA. Prepared by PTI Environmental Services, Inc., Bellevue, WA.

Puget Sound Estuary Protocols (PSEP). 1989a. Recommended protocols for measuring organic compounds in Puget Sound sediment and tissue samples. Final report. Prepared for the U.S. Environmental Protection Agency, Region 10, Office of Puget Sound, Seattle, WA. Prepared by PTI Environmental Services, Inc., Bellevue, WA.

Puget Sound Estuary Protocols (PSEP). 1989b. Recommended protocols for measuring metals in Puget Sound sediment and tissue samples. Final report. Prepared for the U.S. Environmental Protection Agency, Region 10, Office of Puget Sound, Seattle, WA. Prepared by PTI Environmental Services, Inc., Bellevue, WA.

Sturgis, T.C. 1990. Guidance for contracting biological and chemical evaluations of dredged material. Technical Report D-90-10. Prepared by the Dredging Operations Technical Support program, U.S. Army Corps of Engineers, Waterways Experiment Station, Vicksburg, MS.

U.S Army Corps of Engineers (Corps). 1999. Determination on the suitability of dredged material tested under the East Waterway stage II Project and U.S. Coast Guard slip 36. Memorandum for Record. Seattle District, U.S. Army Corps of Engineers, Seattle, WA.

U.S. Environmental Protection Agency (EPA)/U.S Army Corps of Engineers (Corps). 1998. Evaluation of dredged material proposed for discharge in waters of the U.S. – Testing Manual. EPA-823-B-98-004, Washington, DC.

United States Army Corps of Engineers (Corps). 1998. Draft dredged material evaluation framework: Lower Columbia River Management Area. April 1998.

United States Army Corps of Engineers (Corps). 1993. QA/QC guidance for laboratory dredged material bioassays (draft report). Prepared by the Corps' Waterway Experiment Station (WES), Vicksburg, MS.

U.S. Environmental Protection Agency (EPA). 1988a. U.S. Contract Laboratory Program statement of work for organic analyses, multi-media, multi-concentration. U.S. Environmental Protection Agency, Washington DC.

U.S. Environmental Protection Agency (EPA). 1988b. Laboratory Data Validation: Functional guidelines for evaluating organic analyses. U.S. Environmental Protection Agency, Washington DC.

U.S. Environmental Protection Agency (EPA). 1988c. Laboratory Data Validation: Functional guidelines for evaluating inorganic analyses. U.S. Environmental Protection Agency, Washington DC.

U.S. Environmental Protection Agency (EPA). 1994. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. EPA-600-R-94-024, Washington, DC.

U.S. Environmental Protection Agency (EPA). 1993. U.S. Contract Laboratory Program statement of work for inorganic analyses, multi-media, multi-concentration. Document ILMO3.0. U.S. Environmental Protection Agency, Washington DC.

U.S. Environmental Protection Agency (EPA). 1990. Manual for the evaluation of laboratories performing aquatic tests. U.S. Environmental Protection Agency, Washington DC.

U.S. Environmental Protection Agency (EPA). 1990. Manual for the evaluation of laboratories performing aquatic tests. U.S. Environmental Protection Agency, Washington, DC.

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Figure C-1

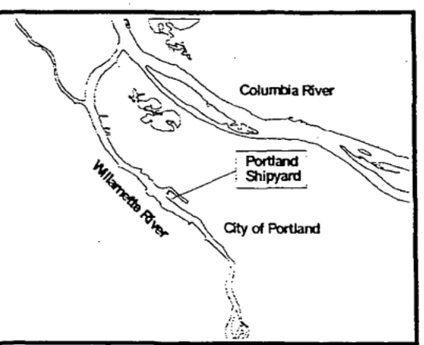
Surface Sediment Sample Locations at Portland Shipyard

Dry Dock 1
Berth 301
Berth 309
Berth 310
PSY(2)-86
PSY(2)-85
Berth 302
PSY(2)-93
Dry Dock 4
Dry Dock 3
PSY(2)-88
PSY(2)-87
PSY(2)-84
PSY(2)-83
PSY(2)-82
PSY(2)-81
PSY(2)-92
Berth 303
PSY(2)-80
PSY(2)-91
Berth 312
Berth 304
Swan Island Lagoon
Willamette River
Berth 313
PSY(2)-94
PSY(2)-95
PSY(2)-96
PSY(2)-97
Portland Shipyard
Berth 305
Berth 311
PSY(2)-78
PSY(2)-79
Berth 306
Berth 307
Berth 308
Berth 314
Berth 315

500 0 500 1000 Feet

LEGEND

- ⊙ Surface Sample
- ⊙ Surface and Core Sample



Portland Shipyard Remedial Investigation	January 2000	Z:\B99-13 PSY 2000\PSY 2000 SAPP_QAPP Figure C-1 PortlandShipyardSurface.cdr

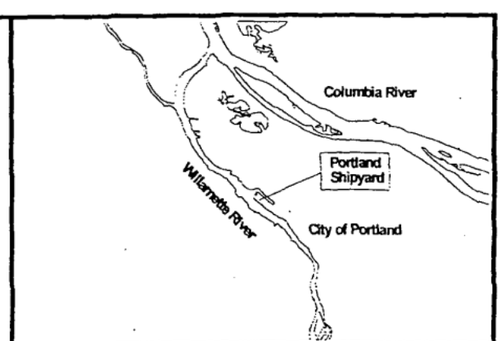
Figure C-2

Subsurface Sediment Sample Locations at Portland Shipyard

Dry Dock 1
Berth 301
PSY(2)-85
Berth 309
Berth 310
Dry Dock 4
PSY(2)-93
Dry Dock 3
PSY(2)-87
PSY(2)-89
PSY(2)-90
PSY(2)-91
Berth 312
Berth 313
Berth 314
Berth 315
Berth 302
PSY(2)-83
PSY(2)-82
Berth 303
Berth 304
Berth 305
Berth 306
Berth 307
Berth 308
Berth 311
PSY(2)-78
Swan Island Lagoon
Portland Shipyard
Willamette River

LEGEND

- Core Sample
- Surface and Core Sample



Portland Shipyard Remedial Investigation

January 2000

Z:\B99-13 PSY 2000\PSY 2000 SAPP_QAPP Figure C-2 PortlandShipyardSubsurface.cdr

SAMPLE DESCRIPTION LOG

SURVEY ________ STATION ________ SEGMENT ________ DATE ____________

CREW ____

RADAR COORDINATES :

____

REPLICATE NO: _____ TIME: ________ COORDINATES : ____________________

BIO/CHEM : Bottom Depth ________ Penetration Depth: ________ RPD Depth ________

SEDIMENT TYPE : Cobble Gravel Sand C M F Silt Clay Wood/Shell Fragments

LARGE FAUNA : Pachycerianthus Phyllochaetopterus Ptilosarcus Crabs Asteroids

SEDIMENT COLOR : D.O. Gray Black Brown Brown Surface

SEDIMENT ODOR : H_2S Petroleum None Slight Moderate Strong Overwhelming

COMMENTS :

____

REPLICATE NO: _____ TIME: ________ COORDINATES : ____________________

BIO/CHEM : Bottom Depth ________ Penetration Depth: ________ RPD Depth ________

SEDIMENT TYPE : Cobble Gravel Sand C M F Silt Clay Wood/Shell Fragments

LARGE FAUNA : Pachycerianthus Phyllochaetopterus Ptilosarcus Crabs Asteroids

SEDIMENT COLOR : D.O. Gray Black Brown Brown Surface

SEDIMENT ODOR : H_2S Petroleum None Slight Moderate Strong Overwhelming

COMMENTS :

____

REPLICATE NO: _____ TIME: ________ COORDINATES : ____________________

BIO/CHEM : Bottom Depth ________ Penetration Depth: ________ RPD Depth ________

SEDIMENT TYPE : Cobble Gravel Sand C M F Silt Clay Wood/Shell Fragments

LARGE FAUNA : Pachycerianthus Phyllochaetopterus Ptilosarcus Crabs Asteroids

SEDIMENT COLOR : D.O. Gray Black Brown Brown Surface

SEDIMENT ODOR : H_2S Petroleum None Slight Moderate Strong Overwhelming

COMMENTS :

____

REPLICATE NO: _____ TIME: ________ COORDINATES : ____________________

BIO/CHEM : Bottom Depth ________ Penetration Depth: ________ RPD Depth ________

SEDIMENT TYPE : Cobble Gravel Sand C M F Silt Clay Wood/Shell Fragments

LARGE FAUNA : Pachycerianthus Phyllochaetopterus Ptilosarcus Crabs Asteroids

SEDIMENT COLOR : D.O. Gray Black Brown Brown Surface

SEDIMENT ODOR : H_2S Petroleum None Slight Moderate Strong Overwhelming

COMMENTS :

____

Figure C-3

Surface Sample Description Log

Field Log by:	Processing by:	Coring by:
Tide Level from MLLW:	Date:	Total Drive Length:
Depth to Mudline:	Time:	Recovered Length:
Mudline Elev.:		Recovery Efficiency:

Note: All elevations, depths, and distances in feet.

Core Description - Core Tube Lengths

Tube Length	Sample No	Visual Description
_1		
_2		
_3		
_4		
_5		
_6		
_7		
_8		
_9		
_10		
_11		
_12		
_13		
_14		
_15		
_16		
_17		
18		

In-Situ Summary Log

Interpreted Summary	Sample No.	Acquisition Notes
_1		
_2		
_3		
_4		
_5		
_6		
_7		
_8		
_9		
_10		
_11		
_12		
_13		
_14		
_15		
_16		
_17		
18		

Core Tube Field Cut Information

Sample No	Tube Length Interval	Segment Length

Sample Test Information

Sample No /Tests	In-Situ Depth Int

Notes:

PRIVILEGED AND CONFIDENTIAL: Work Product Prepared Under Attorney/Client Privilege

Figure C-4

Core Description Log

Portland Shipyard SAP/QAPP

January 2000

PSY-SQ Figure C-4.xar

Table C-1. Surface Station Coordinates (NAD 1927).

STATION	X	Y
PSY(2)-96	7632401.343	699690.2065
PSY(2)-97	7632476.044	699695.6631
PSY(2)-95	7632434.696	699729.0168
PSY(2)-94	7632398.04	699757.9685
PSY(2)-93	7631279.79	700934.6775
PSY(2)-86	7632770.26	701138.5566
PSY(2)-85	7633065.542	701054.1895
PSY(2)-88	7632553.927	700852.3872
PSY(2)-87	7633031.488	700701.3391
PSY(2)-91	7632097.666	700311.4376
PSY(2)-92	7631610.222	700688.6086
PSY(2)-78	7636201.139	699528.5587
PSY(2)-79	7635575.423	699409.0423
PSY(2)-80	7634485.699	700305.4349
PSY(2)-83	7633965.234	700819.3413
PSY(2)-81	7633971.014	700751.5046
PSY(2)-84	7633892.384	700813.7782
PSY(2)-82	7633932.035	700783.6958

Table C-2. Subsurface Station Coordinates (NAD 1927).

STATION	X	Y
PSY(2)-93	7631279.79	700934.6775
PSY(2)-85	7633065.542	701054.1895
PSY(2)-87	7633031.488	700701.3391
PSY(2)-89	7632766.741	700547.9942
PSY(2)-90	7632422.245	700456.599
PSY(2)-91	7632097.666	700311.4376
PSY(2)-78	7636201.139	699528.5587
PSY(2)-83	7633965.234	700819.3413
PSY(2)-82	7633932.035	700783.6958

Table C-3. Target Analytes and Project Required Quantitation Limits (QLs).

Analytes	Required Quantitation Limit
Metals Total (mg/kg dry wt.)	
(EPA 3050, 6010, 7000)	
Antimony	20
Arsenic	57
Cadmium	0.96
Chromium	270
Copper	81
Lead	66
Mercury	0.21
Nickel	140
Silver	1.2
Zinc	160
ButylTins as Ion (ug/l)	
(GC/MS)	
Tributyltin (ug/l interstitial water)	0.05
(GC FPD)	
Bulk Butyltin (ug/kg dry weight)	1
Conventional Analytes	
Total Solids (%)	Std.CAS
Ammonia in Interstitial Water (mg/l)	Std.CAS
Grain Size - ASTM-D-442-63/PSEP	Std.CAS
Volatile Solids (% Not Analyzed)	Std.CAS
TOC (% Dry Weight)	Std.CAS
Semivolatiles organics (ug/kg)	
(EPA 8270)	
Acenapthene	20
Acenapththylene	20
Anthracene	20
Flourene	20
2-Methylnaphthalene	20
Phenanthrene	20
Naphthalene	20
Benzo (a) anthracene	20

Table C-3. Target Analytes and Project Required Quantitation Limits (QLs).

Analytes	Required Quantitation Limit
Benzo (a) pyrene	20
Benzo (b) fluoranthene	20
Benzo (k) fluoranthene	20
Benzo (g, h, I) perylene	20
Chrysene	20
Dibenz (a, h) anthracene	20
Fluoranthene	20
Indeno (1, 2, 3-cd) pyrene	20
Pyrene	20
bis(2-Ethylhexyl)phthalate	20
Dibenzofuran	20
2,4-Dimethylphenol	20
Pentachlorophenol	40
4-Methylphenol	20
Volatile Organics:	
EPA 8260)	
Acetone	10
Chlorobenzene	5
Ethylbenzene	5
Xylenes	5
Benzene	5
Trichloroethylene	5
Tetrachloroethylene	5
Pesticides (ug/kg):	
(EPA 8081)	
Aldrin	10
alpha-BHC	10
beta-BHC	10
delta-BHC	10
gamma-BHC (Lindane)	10
Chlordane	10
4,4'-DDD	6.9
4,4'DDE	6.9

Table C-3. Target Analytes and Project Required Quantitation Limits (QLs).

Analytes	Required Quantitation Limit
4,4'-DDT	6.9
Dieldrin	10
Endosulfan	10
Endosulfan II	10
Endosulfan Sulfate	10
Endrin	10
Endrin Aldehyde	10
Endrin Ketone	10
Heptachlor	10
Heptachlor Epoxide	10
Methoxychlor	20
Toxaphene	
PCBs (ug/kg)	
(EPA 8082)	
Aroclor 1016	33
Aroclor1221	66
Aroclor1232	33
Aroclor1242	33
Aroclor1248	33
Aroclor1254	33
Aroclor1260	33
Total PCB	

Table C-4. Analytical Test Methods and Laboratory QC Criteria.

Analyte	Matrix	Method	Recovery Criteria		
Metals			LCS[1]	MS	
Sb	Soil/Sed	SM-200.8	12.2-90.1	30-120	
As	Soil/Sed	SM-200.8	43.9-81.5	60-130	
Cd	Soil/Sed	SM-200.8	51.4-103	60-130	
Cr	Soil/Sed	SM-200.8	59.4-94.6	60-130	
Cu	Soil/Sed	SM-200.8	45.9-70.4	60-130	
Ni	Soil/Sed	SM-200.8	122-204	60-130	
Ag	Soil/Sed	SM-200.8	51.4-87.7	60-130	
Zn	Soil/Sed	SM-200.8	84.1-144	60-130	
Pb	Soil/Sed	SM-200.8	82.7-160	60-130	
Hg	Soil/Sed	SW-7471A	1.60-3.41	60-130	
Metals-SEM					
Cd	Soil/Sed	SM-6010A	85-115	60-130	
Cu	Soil/Sed	SM-6010A	85-115	60-130	
Ni	Soil/Sed	SM-6010A	85-115	60-130	
Zn	Soil/Sed	SM-6010A	85-115	60-130	
Pb	Soil/Sed	SW-7421	85-115	60-130	
Hg	Soil/Sed	SW-7471	85-115	60-130	
TOC	Soil/Sed	ASTM D4129-82M	85-115	75-125	
AVS	Soil/Sed	Draft Aug. '91	85-115	75-125	
Grain Size	Soil/Sed	PSEP	NA	NA	
Ammonia	Pore W.	160.2	85-115	75-125	
HCID	Soil/Sed	NWTPH	Surrogate		
			o-Terphenyl		50-150
			4-Bromofluorobenzene		50-150
			n-Triacontane		50-150
			LCS		
			Diesel		50-150[2]
			Lube Oil		

Table C-4. Analytical Test Methods and Laboratory QC Criteria.

Analyte	Matrix	Method	Recovery Criteria	
Pesticides	Soil/Sed	SW-8081	Surrogate	
			Tetrachloro-m-xylene	26-116
			Decachlorobiphenyl	33-143
			LCS	
			gamma-BHC (Lindane)	40-124
			Heptachlor	40-117
			Aldrin	43-108
			Dieldrin	46-127
			Endrin	46-123
			4,4'-DDT	46-127
Pesticides (Continued)	Soil/Sed	SW-8081	Matrix Spike	
			gamma-BHC (Lindane)	28-123
			Heptachlor	36-115
			Aldrin	33-114
			Dieldrin	35-126
			Endrin	39-130
			4,4'-DDT	36-135
PCBs	Soil/Sed	SW-8082	Surrogates	70-130
			LCS	70-130
			MS	70-130

Table C-4. Analytical Test Methods and Laboratory QC Criteria.

Analyte	Matrix	Method	Recovery Criteria	
SIM-SVO	Soil/Sed	CAS SOP	Surrogates	
			2-Fluorophenol	31-106
			Phenol-d6	37-104
			2,4,6-Tribromophenol	12-116
			Nitrobenzene-d5	22-123
			2-Fluorobiphenyl	15-117
			p-Terphenyl-d14	19-140
			LCS	
			Phenol	21-110
			2-Chlorophenol	26-113
			1,4-Dichlorobenzene	37-93
			N-Nitrosodi-n-propylamine	19-136
			1,2,4-Trichlorobenzene	10-108
			4-Chloro-3-methylphenol	13-109
			Acenaphthene	29-109
			4-Nitrophenol	10-151
			2,4-Dinitrotoluene	23-136
			Pentachlorophenol	10-120
			Pyrene	39-149
			Matrix Spikes	
			Phenol	20-99
			2-Chlorophenol	17-98
			1,4-Dichlorobenzene	10-109
			N-Nitrosodi-n-propylamine	12-135
			1,2,4-Trichlorobenzene	21-100
			4-Chloro-3-methylphenol	15-113
			Acenaphthene	26-104
			4-Nitrophenol	26-149
			2,4-Dinitrotoluene	10-186
			Pentachlorophenol	10-145
			Pyrene	18-144

Table C-4. Analytical Test Methods and Laboratory QC Criteria.

Analyte	Matrix	Method	Recovery Criteria	
Volatiles	Soil/Sed	SW-8260	Surrogate	
			Dibromofluoromethane	75-132
			Toluene-d8	85-109
			4-Bromofluorobenzene	49-131
			LCS	
			1,1-Dichloroethene	73-118
			Benzene	78-116
			Trichloroethene	79-119
			Toluene	77-118
			Chlorobenzene	80-117
			1,2-Dichlorobenzene	79-120
			Naphthalene	57-135
			Matrix Spike	
			1,1-Dichloroethene	51-127
			Benzene	57-121
			Trichloroethene	45-127
			Toluene	34-134
			Chlorobenzene	37-126
			1,2-Dichlorobenzene	34-131
			Naphthalene	20-139
TBT	Pore W.	Krone 1988	Surrogate	
			Tripropyltin	20-113
			Tripentyltin	20-113
			LCS	20-138
			Matrix Spike	23-127
Bulk TBT	Soil/Sed	CAS SOP	Surrogate	
			Tripropyltin	20-113
			Tripentyltin	20-113
			LCS	20-138
			Matrix Spike	23-127

[1] ppm in ERA/PP CLP Soil Reference Standard

[2] Advisory - NWTPH-HCID does not give LCS criteria

ATTACHMENT C-1
COLUMBIA ANALYTICAL SERVICES
QUALITY ASSURANCE PLAN AND STANDARD OPERATING PROCEDURES

QUALITY ASSURANCE MANUAL

for

Columbia Analytical Services, Inc.
1317 South 13th Avenue
Kelso, Washington 98626

February 5, 1999

Approved by:

Laboratory Director: Jeff Christian

Quality Assurance Manager: Lee Wolf

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sediment, sludge, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities.

CAS maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

The information in this document has been organized according to the format described in *Interim Guidance for the Preparation of Quality Assurance Project Plans*, QAM-005, USEPA, 1980; and *Guidance on Preparation of Laboratory Quality Assurance Plans*, USEPA, February 14, 1991.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

> "The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 25,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types.

These specialized areas include:

- Shipping and Receiving/Purchasing
- Sample Management Office (including a separate, controlled-access sample storage facility)
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry & General Chemistry Laboratories
- Gas Chromatography Laboratory (including a separate sample preparation laboratory)

- Gas Chromatography/Mass Spectrometry Laboratory (including a separate sample preparation laboratory)
- Petroleum Hydrocarbon Laboratory (including a separate sample preparation laboratory)
- Volatile Organics Laboratory (including a separate sample preparation laboratory)
- Microbiology Laboratory
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation is appropriate for the procedures in use. Appendix B lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, QAPPs, and this QA Manual. Documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, and preventative maintenance procedures further ensure the quality of data produced. Performance Evaluation (PE) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PE samples are obtained from qualified vendors and are performed on a regular basis. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas.

The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, the senior laboratory management staff at CAS examine a number of other performance indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, Analytical Report defect rate and Customer Invoice defect rate are a few of the measurements performed at CAS that are used to assess performance from an external perspective (i.e., client satisfaction). The use of these and other indicators is outlined in the *SOP for Nonfinancial Performance Measures* (SOP No. ADM-NFPM). A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload. CAS utilizes a number of different methods to insure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results.

4.4 Document Control

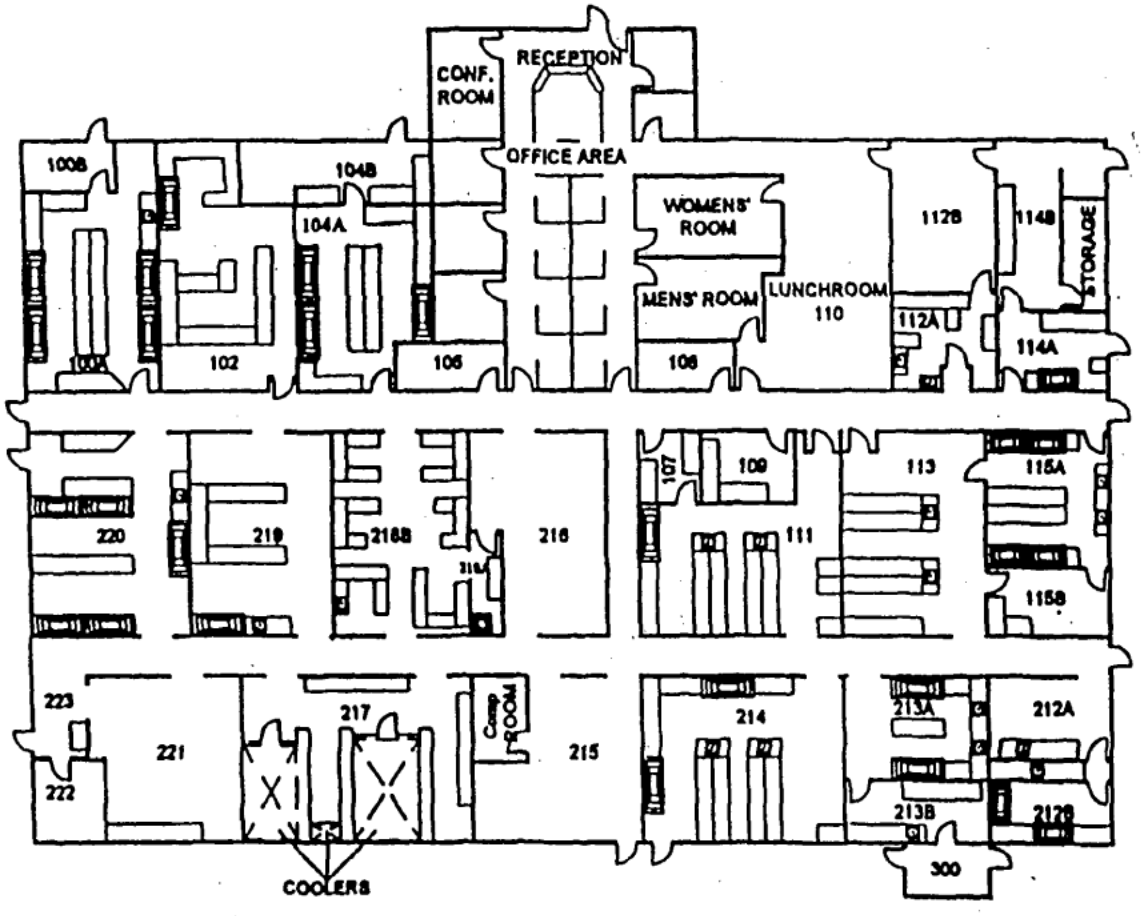
Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), safety manuals (SFM), and other controlled CAS documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution in performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed or is in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

4.5 Subcontracting

Analytical services are subcontracted when CAS/Kelso needs to balance workload and/or when the requested analyses are not performed by CAS/Kelso. However, subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done to capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories Outside of CAS Network (ADM-SUBLAB).*

Figure 4-1
CAS/Kelso Laboratory Floor Plan



Room Designations			
100A Organic Extractions Lab	111 General Chemistry Lab	213A Digestion Lab	221 Bottle Preparation Room
100B Electrical Room	112A Microbiology Lab	213B Sample Preparation	222 Purchasing Room
102 Chromatography Lab	112B Copy Center	214 General Chemistry Lab	223 General Receiving
104A Organic Extractions Lab	113 Atomic Absorption Lab	215 IT Department	300 Janitorial Room
104B Chromatography Lab	114A SMO / Receiving	216 QA/Records	
106 Glasswashing Room	114B SMO / Receiving	217 Stock Room / Sample	
107 Oven Room	115A Metals Digestion Lab	Storage	
108 Zero Headspace Extractions	115B ICP Lab	218A VOA Prepration	
Lab	212A ICP / MS Lab	218B VOA Lab	
109 Sample Preparation	212B Clean Digestion Lab	219 GC/MS Lab	
110 Lunchroom		220 Organic Extraction Lab	

TITLE	CAS Kelso Floor Plans
FILENAME: KFLRPLAN.VSD	PAGE: 1 OF 2
DATE: 9/13/96	DESCRIPTION: Laboratory

Legend

Fume Hood — Sink

Emergency Shower — Sink

5.0 STATEMENT OF PROFESSIONAL CONDUCT AND LABORATORY PRACTICE

One of the most important aspects of the success of CAS as a company is the emphasis placed on the integrity of the data that are provided and the services that are performed. To promote product quality, CAS requires certain standards of conduct and ethical performance among our employees. The following examples of documented CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.

Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.

It is the responsibility of all CAS employees to safeguard sensitive company and client information. The nature of our business and the economic well-being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. No information may be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken very seriously and is subject to formal disciplinary action. As a condition of employment, all employees are required to sign and adhere to confidentiality requirements set forth in CAS' "Employee Agreement" at date of hire and upon termination.

At the time of hire, each employee is also required to sign a Commitment to Excellence in Data Quality. Employees are periodically reminded of their data quality and ethical conduct responsibilities.

6.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 100 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that a modern, state-of-the-art analytical laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the CAS/Kelso personnel assigned to these key positions. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix A.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation the quality program and to coordinate overall QA activities within the laboratory. The QAM works with individual laboratory production units to establish effective quality control and assessment plans. The QAM is also responsible for maintaining this QA Manual and performing an annual review of it, updating it if necessary; reviewing, approving, and controlling SOPs and coordinating the annual review of each SOP (Section 4.2.1); maintaining QA records such as metrological records, archived logbooks, PE sample results, etc.; coordinating PE sample analyses and approving nonconformity and corrective action reports for any unacceptable PE sample results (Section 15.0); reviewing data (Section 12.0); maintaining the laboratory's certifications and approvals (Section 13.0); performing internal QA audits (Section 13.0); preparing QA activity reports (Section 16.0); etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the CAS Quality Assurance Director, who is responsible for the CAS laboratory-wide QA program.

  The Quality Assurance Director is responsible for the overall QA program at all the CAS laboratories. The QA Director is responsible for performing an annual on-site audit at each CAS laboratory and preparing a written report; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a

data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing an annual QA activity report; etc.

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to CAS' EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and marketing functions) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within that department's responsibilities. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each analyst in the laboratory has the responsibility to carry out testing according to prescribed methods, standard operating procedures and quality control guidelines particular to the laboratory in which he/she is working.

- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, education of analytical staff in the use of scientific software, software development and implementation, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	20	Laboratory Director
Lee Wolf, B.S.	13	Quality Assurance Manager
Eileen Arnold, B.A.	18	Environmental, Health and Safety Officer
Lynda Huckestein, B.S.	13	Senior Project Chemist Client Services Manager Sample Management Office Manager
Joe Wiegel, B.S.	9	Organics Manager Semivolatile GC/LC Dept. Manager
Jeff Coronado, B.S.	9	Metals Department Manager
Greg Jasper, A.A.	10	Metals Digestion Supervisor
Todd Poyfair, B.S.	7	General and Water Chemistry Department Manager
Jeff Grindstaff, B.S.	10	Volatiles and Semivolatiles GC/MS Department Manager
David Edelman, Ph.D.	19	CAS Technical Director, CAS Information Technology Director
Lawrence Jacoby, Ph.D.	26	CAS Quality Assurance Director CAS Environmental, Health and Safety Director
Steve Vincent, B.S.	23	CAS President

7.0 SAMPLING, SAMPLE PRESERVATION, AND HANDLING PROCEDURES

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in 40 CFR 136, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sample handling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010 and Supplement I, 600/4-82-057, 600/R-93/100, 600/4-88-039 and Supplements I and II, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. The complete citation for each of these references can be found in Section 18.0 of this document. The container, preservation and holding time information is summarized in Table 7-1.

CAS routinely provides sample containers with appropriate preservatives for our clients. The containers are purchased as "precleaned" to a level 1 status, and conform to the requirements for analytical sample established by the USEPA. Certificates of analysis for the sampling containers are available to clients if requested. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (decontaminated inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. An example of a sample container label and a custody seal is shown in Figure 7-1. Figure 7-2 is a copy of the chain-of-custody form routinely used at CAS. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative will be always be added to the sample containers prior to shipment, unless otherwise instructed by the client. If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination

process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides its own courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When environmental samples are shipped by CAS to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (*Standard Operating Procedure for Chain of Custody for Sample Transfer between Laboratories* [SOP No. ADM - COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

Table 7-1
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Fecal and Total	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity	W	P,G	Cool, 4°C	14 days
Alkalinity	W	P,G	Cool, 4°C	14 days
Ammonia	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (BOD)	W	P,G	Cool, 4°C	48 hours
Bromide	W	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride	W	P,G	None Required	28 days
Chlorine, Total Residual	W	P,G	None Required	24 hours
Color	W	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Fluoride	W	P,G	None Required	28 days
Hardness	W	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH)	W	P,G	None Required	24 hours
Kjeldahl and Organic Nitrogen	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrate	W	P,G	Cool, 4°C	48 hours
Nitrate-Nitrite	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite	W	P,G	Cool, 4°C	48 hours
Orthophosphate	W	P,G	Filter Immediately, Cool, 4°C	48 hours
Oxygen, Dissolved (Probe)	W	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Phenolics, Total	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Elemental	W	G Only	Cool, 4°C	48 hours
Phosphorus, Total	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	W	P,G	Cool, 4°C	7 days
Residue, Settleable	W	P,G	Cool, 4°C	48 hours
Residue, Volatile	W	P,G	Cool, 4°C	7 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Silica	W	P Only	Cool, 4°C	28 days
Specific Conductance	W	P,G	Cool, 4°C	28 days
Sulfate	W	P,G	Cool, 4°C	28 days
Sulfide	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite	W	P,G	None Required	24 hours
Surfactants (MBAS)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin	W	P,G	Cool, 4°C	28 days
Temperature	W	P,G	None Required	Analyze immediately
Turbidity	W	P,G	Cool, 4°C	48 hours
Metals				
Chromium VI	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
	S	P,G	Cool, 4°C	28 days
Metals, except Chromium VI and Mercury	W	P,G	HNO_3 to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Organic Tests				
Oil and Grease	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (TOC)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (TOX)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (AOX)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total Recoverable	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
Petroleum Hydrocarbons, Total	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Purgeable Halocarbons	W	G, Teflon-Lined Septum Cap	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C, No Headspace	14 days
	S	G, Teflon-Lined Cap, or 5035[j]	Cool, 4°C, Minimize Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE)	W	G, Teflon-Lined Septum Cap	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C, No Headspace	14 days
	S	G, Teflon-Lined Cap, or 5035[j]	Cool, 4°C, Minimize Headspace	14 days
Acrolein, Acrylonitrile, Acetonitrile	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
EDB and DBCP	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Alcohols and Glycols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phenols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phthalate Esters	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrosamines	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Nitroaromatics and Cyclic Ketones	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Polynuclear Aromatic Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Haloethers	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids	W	G, Teflon-Lined Cap	NaOH to pH ≥10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction
Semivolatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organochlorine Pesticides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Contract Laboratory Program (CLP)				
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH 12, plus 0.6 g Ascorbic Acid	12 days[h]
	S	P,G	Cool, 4°C	12 days[h]
Mercury	W	P,G	HNO_3 to pH<2	26 days[h]
	S	P,G	Cool, 4°C	26 days[h]
Metals, except Mercury	W	P,G	HNO3 to pH<2	6 months[h]
	S	P,G	Cool, 4°C	6 months[h]
Volatile Organics	W	G, Teflon-Lined Cap	HCl to pH <2, Cool, 4°C, Minimize Headspace	10 days[h]
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	10 days[h]
Semivolatile Organics	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	5 days until extraction;[h,i] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	5 days until extraction;[h,i] 40 days after extraction

a See Section 18.0 for sources of holding time information.
b W = Water, S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass
d For chlorinated water samples
e The recommended maximum holding time is variable, and is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.
h Number of days following sample receipt at the laboratory.
i Ten days until extraction for soil, sediment, and sludge samples.
j For EPA Method 5035, refer to the method for details on sampling and preservation.

Figure 7-1
Sample Container Label and Custody Seal

COLUMBIA ANALYTICAL SERVICES, INC.

Project Name: _____

Sample Name: _____

Date_____ Time_____ Sampler _____

Analysis _____

_____

_____

Preservative: _____

Comments:

1-800-695-7222 Lab Label #2

Custody Seal

Date_____ Project _____

Signature_____ Container#_____ of _____

Figure 7-2
Chain of Custody Form

Columbia Analytical Services, Inc.
An Employee-Owned Company

CHAIN OF CUSTODY

SR#:_____

1317 South 13th Ave. • Kelso, WA 98626 • (360) 577-7222 • (800) 695-7222 • FAX (360) 636-1068

PAGE _____ OF _____ COC #_____

PROJECT NAME
PROJECT NUMBER
PROJECT MANAGER
COMPANY/ADDRESS
PHONE # | FAX #
SAMPLER'S SIGNATURE

SAMPLE I.D.	DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS	Semivolatile Organics by GC/MS 625 ☐ 8270 ☐	Volatile Organics 624 ☐ 8260 ☐	Pesticide / Herbicides 608 ☐ 8081 ☐	Total Toxic Organics 608 ☐	PCB AROCLORs 8082	NWTPH-HCID ☐ NWTPH-D ☐	NWTPH-G ☐	Hydrocarbon Scan ☐	Chlorophenolics 8151M Tri ☐ Tetra ☐	PAHs 8310 ☐	PCP ☐	GC/MS-SIM PAH ☐	SIM ☐	Metals, Total or Dissolved (See list below)	Phthalates ☐	Cyanide ☐	Hex-Chrom ☐	pH, Cond., Cl, SO_4, PO_4, F, NO_2, NO_3, BOD, TSS, TDS (circle)	NH_3-N, COD, Total-P, TKN, TOC, DOC (circle)	TOC 9020 ☐	AOX 1650 ☐	508 ☐					REMARKS

REPORT REQUIREMENTS

___ I. Routine Report: Method Blank, Surrogate, as required
___ II. Report Dup., MS, MSD as required
___ III. Data Validation Report (includes all raw data)
___ IV. CLP Deliverable Report
___ V. EDD

INVOICE INFORMATION

P.O. # _____
Bill To: _____

TURNAROUND REQUIREMENTS

____ 24 hr. ____ 48 hr.
____ 5 Day
____ Standard (10-15 working days)
____ Provide FAX Results

_____
Requested Report Date

Circle which metals are to be analyzed:

Total Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

Dissolved Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

SPECIAL INSTRUCTIONS/COMMENTS:

RELINQUISHED BY:	RECEIVED BY:	RELINQUISHED BY:	RECEIVED BY:
Signature / Date/Time	Signature / Date/Time	Signature / Date/Time	Signature / Date/Time
Printed Name / Firm	Printed Name / Firm	Printed Name / Firm	Printed Name / Firm

8.0 SAMPLE CUSTODY

Standard Operating Procedures have been established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Samples delivered to the CAS sample management office (SMO) and are received by a Sample Custodian. A Cooler Receipt and Preservation Check Form (CRF - See Figure 8-1 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity by the Sample Custodian includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);
- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (No assessment of proper preservation is performed for VOC containers by SMO personnel).

Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and chain of custody documents. All potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When a satisfactory resolution has been reached by the Project Chemist and client, the log-in process may commence and analysis may begin. During the log-in process, each sample is given a unique laboratory code and a service request form is generated. The service request contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. This service request is reviewed by the appropriate Project Chemist for accuracy, completeness, consistency of requested analyses and for client project objectives.

Facility security and access is important in maintaining the integrity of samples received at CAS/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The CAS facility is equipped with an alarm system and CAS employs a private security firm to provide night-time and weekend security.

Samples are kept refrigerated until they undergo analysis, unless otherwise specified. CAS stores samples in one of nine various refrigerators or freezers, depending on the type of analysis and the matrix of the sample. CAS has two walk-in refrigerators which house the majority of sample containers received at the laboratory. In addition to the two walk-in refrigerators, there are three additional refrigerators, including dedicated refrigerated storage of VOC samples. These refrigerators are segregated by matrix type (soil or water) and method of analysis. CAS also has three sub-zero freezers capable of storing samples at -20° C; these are primarily used for tissue and sediment samples requiring specialized storage conditions. One additional freezer provides additional frozen storage capacity for miscellaneous samples. The temperature of each sample storage unit used at CAS is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the two walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

Upon completion of all analyses, aqueous samples and sample extracts are retained at ambient temperature on holding shelves for 30 days (unless other arrangements have been made in advance), and soil samples are retained at ambient temperature on holding shelves for 60 days. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Health and Safety Manual*. It should be noted that all waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with all applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal. This ensures that an accurate history of the sample from "cradle to grave" is generated.

Figure 8-1
Cooler Receipt and Preservation Check Form

Columbia Analytical Services Inc.
Cooler Receipt And Preservation Form

Project/Client_____ Work Order K99_____

Cooler received on_____ and opened on _____ by _____

1.	Were custody seals on outside of cooler? If yes, how many and where?_____	YES	NO
2.	Were seals intact and signature & date correct?	YES	NO
3.	COC # _____ _____ _____ _____ _____		
	Temperature of cooler(s) upon receipt: _____ _____ _____ _____ _____		
	Temperature Blank: _____ _____ _____ _____ _____		
4.	Were custody papers properly filled out (ink, signed, etc.)?	YES	NO
5.	Type of packing material present _____		
6.	Did all bottles arrive in good condition (unbroken)?	YES	NO
7.	Were all bottle labels complete (*i.e.* analysis, preservation, etc.)?	YES	NO
8.	Did all bottle labels and tags agree with custody papers?	YES	NO
9.	*Were the correct types of bottles used for the tests indicated?*	*YES*	*NO*
10.	Were all of the preserved bottles received at the lab with the appropriate pH?	YES	NO
11.	Were VOA vials checked for absence of air bubbles, and if present, noted below?	YES	NO
12.	Did the bottles originate from CAS/K or a branch laboratory? _____	YES	NO

Explain any discrepancies_____

_____

_____

_____

Samples that required preservation or received outside of temperature range at the lab(circle)

Sample ID	Reagent	Volume	Lot Number	Initials

9.0 QUALITY CONTROL OBJECTIVES (PRECISION, ACCURACY, AND MDLS)

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated in its laboratories. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from control-charting the various QC measurements for each analytical method. The Quality Control objectives are defined below and the numeric values are shown in the table in Appendix C.

9.1 Accuracy

Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of standard reference materials, standard solutions and laboratory-fortified blanks. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value.

If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). CAS utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

9.2 Precision

Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

At CAS, our "within-batch" precision is measured through the use of replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the replicate measurements. The "batch-to-batch" precision is calculated from the variance observed in results from analysis of standard solutions or laboratory control samples from multiple analytical batches.

9.3 Control Limits

The acceptance limits for accuracy and precision (shown in the table in Appendix C) originate from two different sources: For analyses having enough QC data, control limits are calculated at the 99% confidence limits. New control limits are generated using the data generated in the previous year. After review of the data by the Quality Assurance Manager, the new acceptance criteria replace the previous criteria and method conformity is assessed using the new values. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have control limits stated in it, then control limits are assigned reasonable values. These control limits are updated when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change.

The acceptance limits for accuracy and precision shown in the table in Appendix C are given for the following QC samples: For accuracy limits, the values listed are for laboratory control samples. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

9.4 Representativeness

Representativeness is the degree to which the field sample represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed gives results identical to analysis of the entire field sample. CAS has sample handling procedures and protocols to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Sample Preparation, Compositing, and Subsampling*, the *SOP for Solid Sample Preparation*, and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

9.5 Completeness

Completeness is a measure of the amount of valid data that is obtained, compared to the amount that is expected. For the purposes of this plan, completeness is calculated by dividing the number of samples having valid data by the total number of samples in the project, expressed as a percentage. The CAS objective for completeness is 100%.

9.6 Comparability

Comparability expresses the confidence with which one data set can be compared to another. To ensure comparability, standard operating procedures are used for the preservation, handling, and analysis of all samples. Data is reported in units specified by the customer.

9.7 Method Detection Limits

Method Detection Limits (MDL) for analytical methods routinely performed at CAS/Kelso are determined annually. The MDLs are determined by following the *Standard Operating Procedure for the Determination of Method Detection Limits* (SOP No. ADM - MDL) which is based on the procedure outlined in 40 CFR Part 136, Appendix B. The Method Reporting Limits (MRLs) used at CAS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which CAS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

10.0 QUALITY CONTROL PROCEDURES

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes which may impact analytical results are also described below.

10.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS maintains a database of SOPs for use in both technical and administrative functions. SOPs are written following the format and content requirements described in the *SOP for Preparation of Standard Operating Procedures* (SOP No. ADM-SOP). Each SOP has been reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. A comprehensive list of current SOPs is maintained by the QA Manager. The document control process ensures that only the most currently prepared version of an SOP is being used for guidance and instruction. The QA Manual, QAPPs, SOPs, standards preparation logbooks, run logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (SOP No. ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the promulgated methodology used to perform analyses. Laboratory notebook entries have been standardized following the guidelines in the *Making Entries into Logbooks and onto Benchsheets* SOP (SOP No. ADM-DATANTRY). The entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval (e.g., weekly, monthly, etc.).

10.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the standard operating procedure by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority.

Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the possible need for a change in policy will be addressed by the laboratory director.

10.3 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

10.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that CAS has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner.

The minimum requirements of an analytical batch are:

1. The number of (field) samples in a batch is not to exceed 20.

2. All (field) samples in a batch are of the same matrix.

3. The QC samples to be processed with the (field) samples include:

   a. Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b. Laboratory Control Sample (a.k.a. Laboratory Fortified Blank)

      Function: Assessment of method performance

   c. Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)

      Function: Assessment of matrix problems

      NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked.

d. Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)

Function: Assessment of batch precision

NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be duplicated.

4. A single lot of reagents is used to process the batch of samples.

5. Each operation within the analysis is performed by a single analyst/technician/chemist, or by a team of analysts/technicians/chemists.

6. The time frame is not to exceed a 24 hour period. "Open batches" extending over more than one 24 hour period are not allowed.

7. (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8. The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9. Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

10. Specific project, program, or method SOP requirements may be exceptions. If project, program, or method SOP requirements are more stringent than these laboratory minimum requirements, then the project, program, or method SOP requirements will take precedence. However, if the project, program, or method SOP requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

10.5 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Reporting Limit

(MRL) or, if required, the Method Detection Limit (MDL) for the analyte(s) being tested, otherwise, corrective action must be taken. A method blank is included with the analysis of every analytical batch, every 20 samples, or as stated in the method, whichever is more frequent.

10.6 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve.

10.7 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater.

10.8 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard solutions which are, in turn, prepared from stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

10.9 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity of the standards used in the calibration. Once it is determined that there is no systematic error in preparation of the calibration standard(s), they are considered valid standards and may be used for subsequent calibrations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards. ICVs are also analyzed in accordance with method-specific requirements.

10.10 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

10.11 Internal Standards

Internal standards consist of known amounts of specific compounds that are added to each sample following sample preparation or extraction. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by certain matrix effects. The integrated area of the internal standard compared to the continuing calibration check standard should vary by no more than the limits specified in each method.

10.12 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance. The acceptance criteria for these various analytes are listed in Appendix C, along with other data quality capabilities.

10.13 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) has been added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. The stock solutions used for spiking the sample(s) are purchased and prepared independently of calibration standards. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

For the appropriate methods, matrix spiked samples are prepared and analyzed at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples.

10.14 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

Relative Percent Difference (RPD) = (S1 - S2) x 100 ÷ S_{ave}

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples.

10.15 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks or Quality Control Samples)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free soil (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is(are) added. A standard reference material (SRM) of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. The LCS sample is prepared and analyzed in the same analytical batch, and in exactly the same manner, as the other routine samples. Stock solutions used for LCSs are purchased or prepared independently of calibration standards. The percent recovery (% REC.) of the target analytes in the LCS assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy. Acceptance criteria for LCS analyses are obtained through the use of control charts. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent.

If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

10.16 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The ICS is spiked with the elements of interest at concentrations of approximately ten times the instrument detection limits. The ICS is analyzed at the beginning and end of an analytical run or every eight hours, whichever is more frequent, and the results must be within ± 20% of the true values.

10.17 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the instrument detection limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

10.18 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a Standard Reference Material (SRM). Metrology equipment (analytical balances, thermometers, etc.) is calibrated using SRMs traceable to the National Institute of Standards and Technology (NIST). These primary SRMs are themselves recertified on an annual basis. All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type (see Section 7.0). This information is provided to the client when requested.

Consumable SRMs routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally-recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. CAS relies on a primary vendor for the majority of its analytical supplies; the selection of this vendor is made following the guidelines in the *Primary Vendor Process* SOP, (SOP No. ADM-PVP). In addition, consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, E. M. Science, etc. are examples of the vendors used by CAS. All reference materials that are received at CAS are recorded by the technical staff in the appropriate notebook(s) and are stored under conditions that provide maximum protection against deterioration and contamination. The notebook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. In addition, the date that the standard is received in the laboratory is marked on the container. When the SRM container is used for the

first time, the date of usage and the initials of the applicable technician are also recorded on the container. Stock solutions and/or calibration standard solutions are prepared fresh as often as necessary according to their stability. After preparation, all standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to introduction into the analytical system/process, **all** reference materials are verified with a second, independent source of the material (see section 10.9 above). Once the reference material has been verified to be accurate, it may then be used for instrument calibration and subsequent quantitative purposes. In addition, the independent source of reference material is also used to check the calibration standards for signs of deterioration.

10.19 Control Charting

The generation of control charts is routinely performed at CAS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts which have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements.

10.20 Glassware Washing

Glassware washing and maintenance play an crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

11.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below.

11.1 Temperature Control Devices

Temperatures are monitored and recorded for all of our temperature-regulating devices including ovens, incubators and refrigerators. Bound record books are kept which contain daily recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the appropriate SOP (SOP No. SMO-DALYCK). All thermometers have been identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

11.2 Analytical Balances

Analytical balances are serviced on an semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on an annual basis. The calibration of each analytical balance is checked daily with three class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high ranges. As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Bound record books are kept which contain the recorded measurements, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the appropriate SOP (SOP No. SMO-DALYCK).

11.3 Water Purification System

The water purification system used at CAS is designed to produce deionized water of 18 megohms resistivity or better, meeting specifications for ASTM Type I water. The system is monitored continuously with an on-line meter, which is recorded daily in a bound record book. Deionizers are rotated and replaced when the first unit in the series produces water of 0.5 megohms, which is monitored by a light on the unit. The status of the deionizers is

also checked (resistivity reading and light status) and recorded daily in a bound record book. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. Finally, the water is checked at a point downstream of the original source - typically a spigot in one of the laboratory operating units. This information is also recorded on a weekly basis.

11.4 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the EPA CLP Statement of Work for Inorganic Analysis, SOW No ILM04.0.

11.5 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

11.6 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples.

11.7 GC/MS Systems

All GC/MS instruments are calibrated at a minimum five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All SRMs used for this function are "EPA-Certified" and/or "A_2LA-Certified" standards. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed.

11.8 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. Initial calibration standards are prepared at three to five concentration levels for each analyte of interest. The lowest standard is near the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples.

11.9 Infrared Analyzer/FTIR

The instrument is calibrated using a blank and four standards. The calibration is validated at the beginning of each analysis, and continuing calibration is verified at a minimum frequency of once every ten samples.

11.10 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at CAS include total phenolics, phosphates, surfactants and tannin-lignin.

11.11 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of three standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and five (or six) standards are used to calibrate the instrument for all other automated chemistries. Standard CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

11.12 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a 5-point calibration curve. A correlation coefficient of 0.995 or better for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and bromide.

11.13 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

11.14 Ion-selective electrode

Two standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

11.15 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. Autopipet calibration is verified each day of use. The results of all calibration verifications are recorded in bound logbooks.

11.16 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy version of the data is forwarded to the supervisor or the department manager, who reviews the data for errors and manually rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or senior laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival.

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This includes polychlorinated biphenyls and hydrocarbon fuels (e.g., gasoline and diesel).

- The sample is analyzed for benzene, toluene, ethylbenzene, xylenes, and naphthalene (BTEXN), and the sample is found, by a separate analysis, to contain gasoline. In a sample containing no gasoline, the presence of BTEXN compounds will be confirmed.

- The sample meets all of the following requirements:

    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out, and the documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods

    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).

    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.

- GC/MS Methods - Two criteria are used to verify identification:

    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.

    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Validation

The integrity of the data generated in the laboratory is assessed through the evaluation of the results of the analysis of method blanks, laboratory control samples, sample duplicates, matrix spiked samples, QC samples, trip blanks, et al. The numerical criteria for evaluation of these QC samples are listed within each method-specific Standard Operating Procedure. These various QC sample analyses are evaluated using the flow diagrams found in Figures 12-1 through 12-9. Other validation measures of the data include a check of the linearity of the calibration curve, an accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

12.5 Documentation

12.5.1 Documentation and Archiving of Routine Analysis Data

The archiving system includes all of the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate);
- Instrument parameters;
- Sample analysis sequence;
- Analysis benchsheets and instrument printouts;
- Chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Log book ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Report (NCAR) forms, if necessary.

Individual sets of analyses are indexed by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed,

they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

12.5.2 Documentation of QC Data

To summarize the recovery data for surrogates and matrix spikes, a separate documentation system has been established. The results are segregated according to the sample matrix. Additional information is included, indicating those results affected by matrix interferences, etc. Surrogate and matrix spike acceptance limits are listed in Appendix C. This system also includes results for the most recent calibration curves, as well as method validation results.

12.5.3 Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Basic specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications.

Figure 12-1
Evaluation of Method Calibration

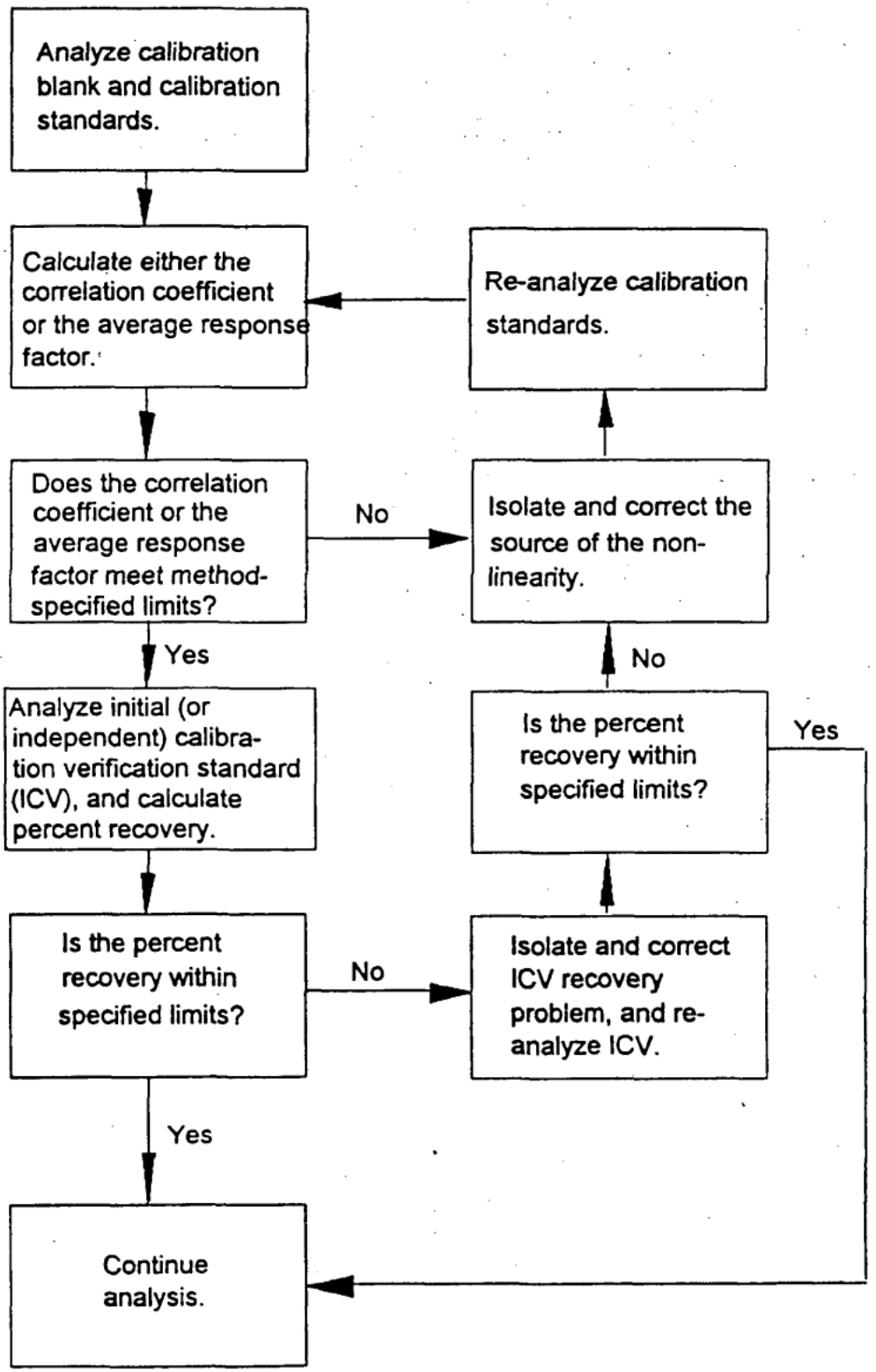


Figure 12-2
Evaluation of Continuing Calibration

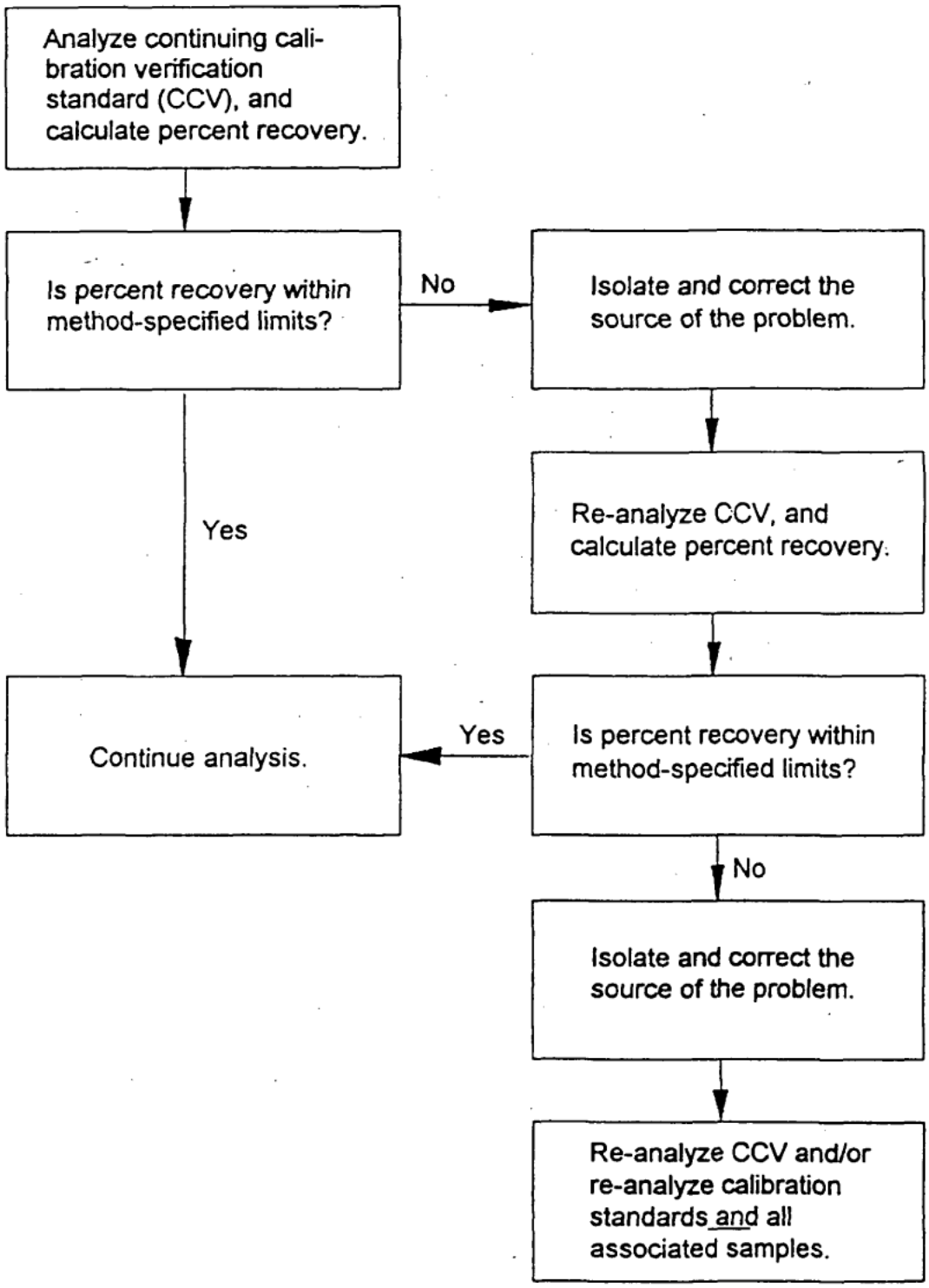


Figure 12-3
Evaluation of Method Blank and Instrument Blank Results

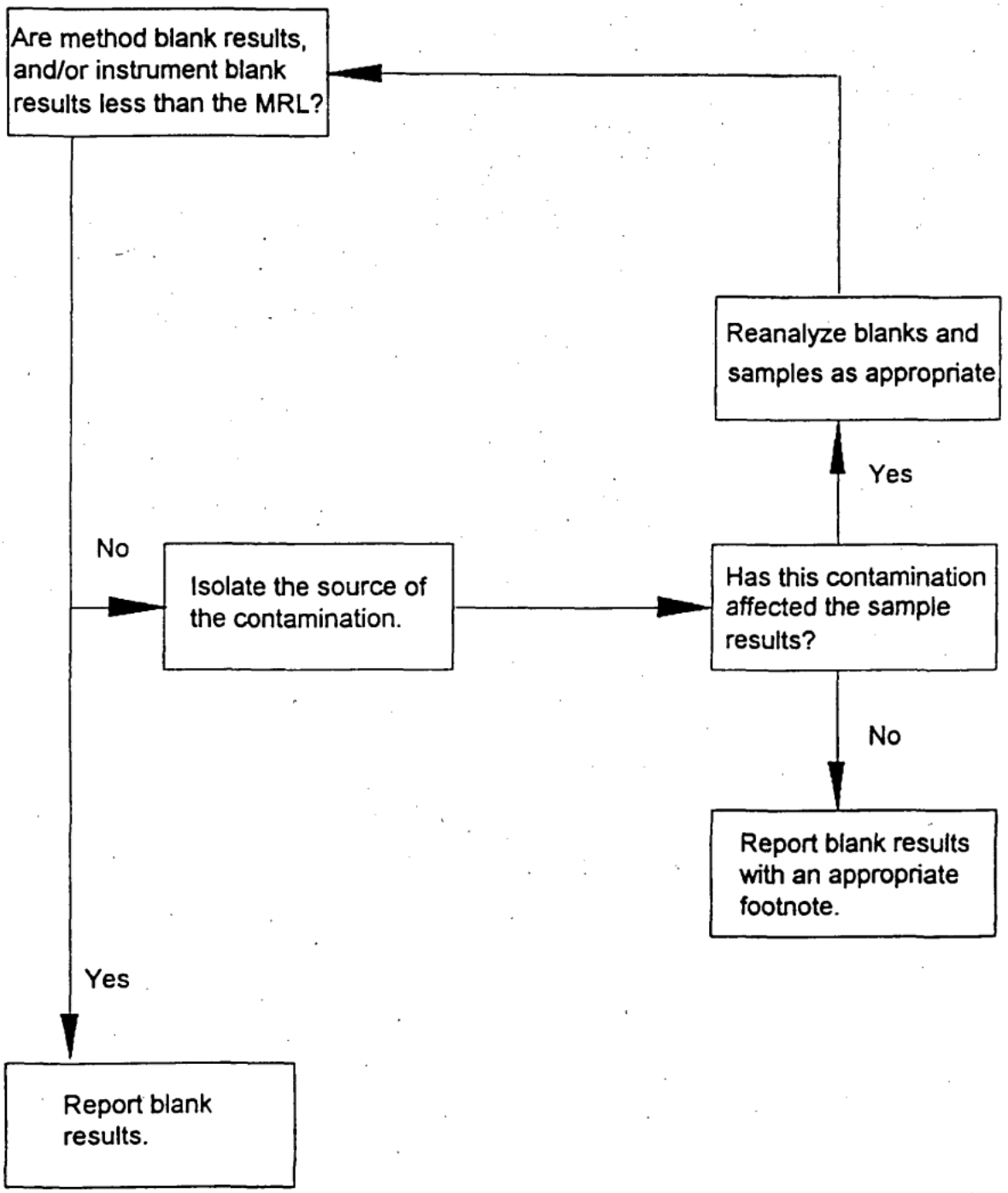


Figure 12-4
Evaluation of Sample Results for Inorganic Analyses

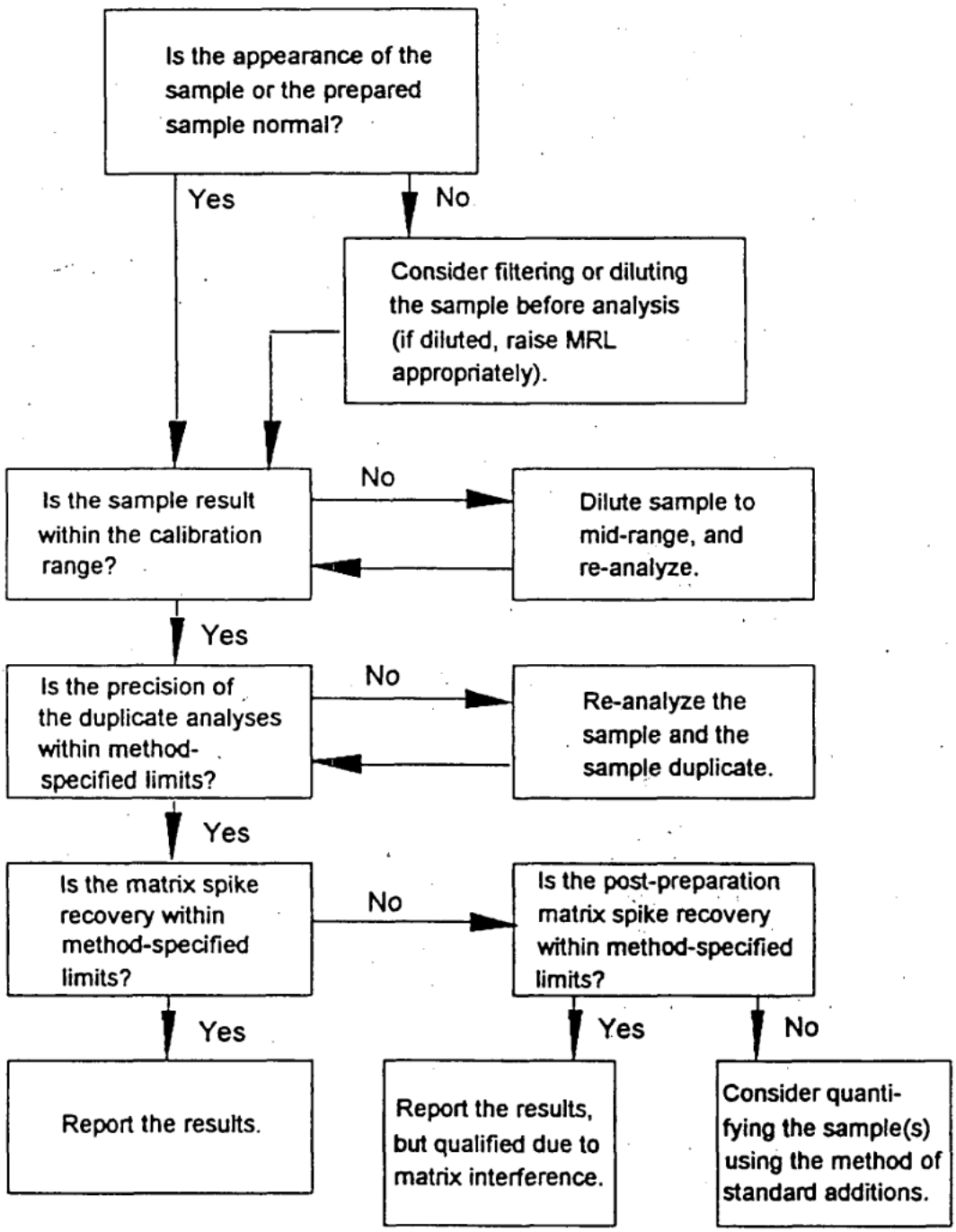


Figure 12-5
Evaluation of Sample Results for Organic Analyses

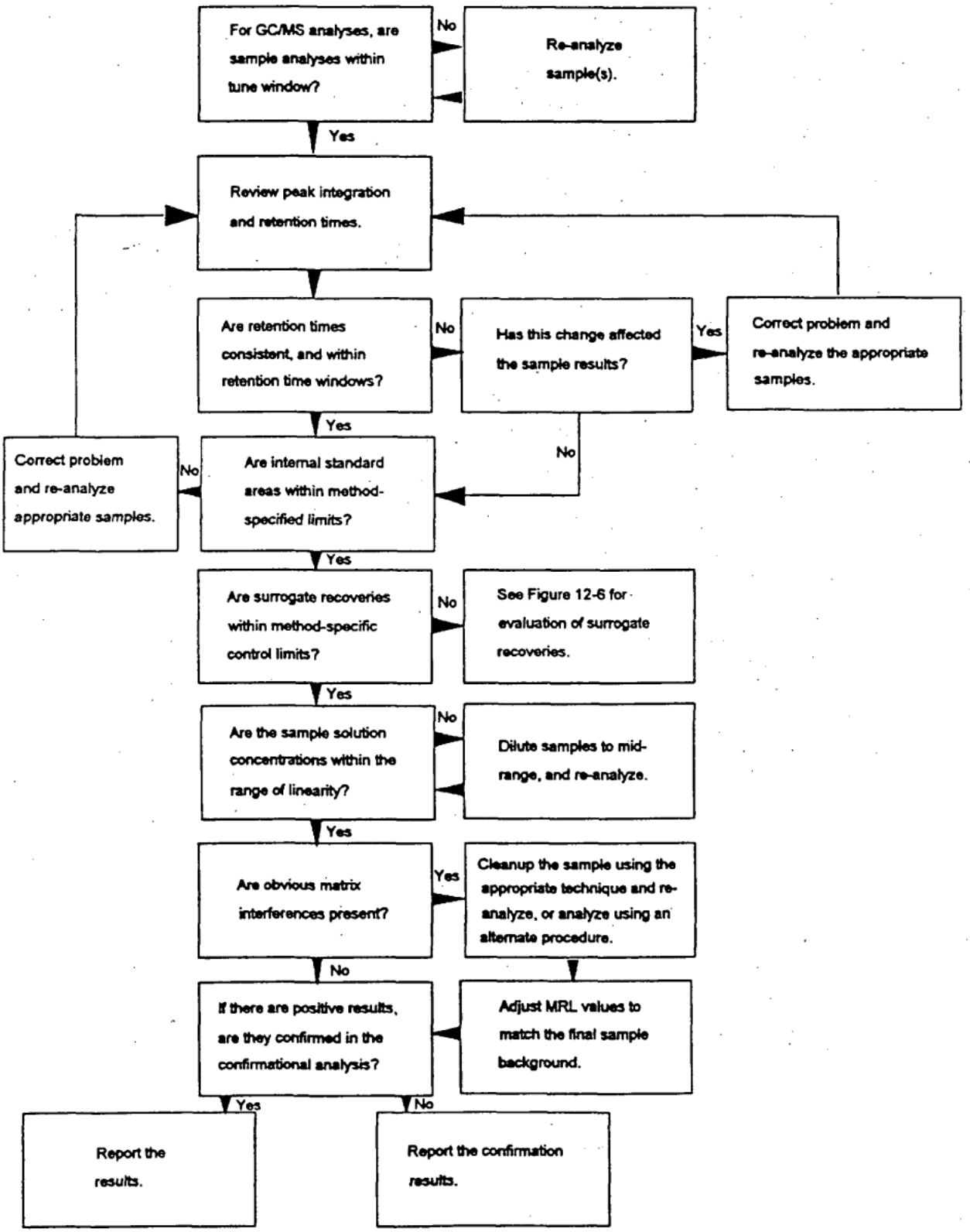


Figure 12-6
Evaluation of Surrogate Compound Recoveries

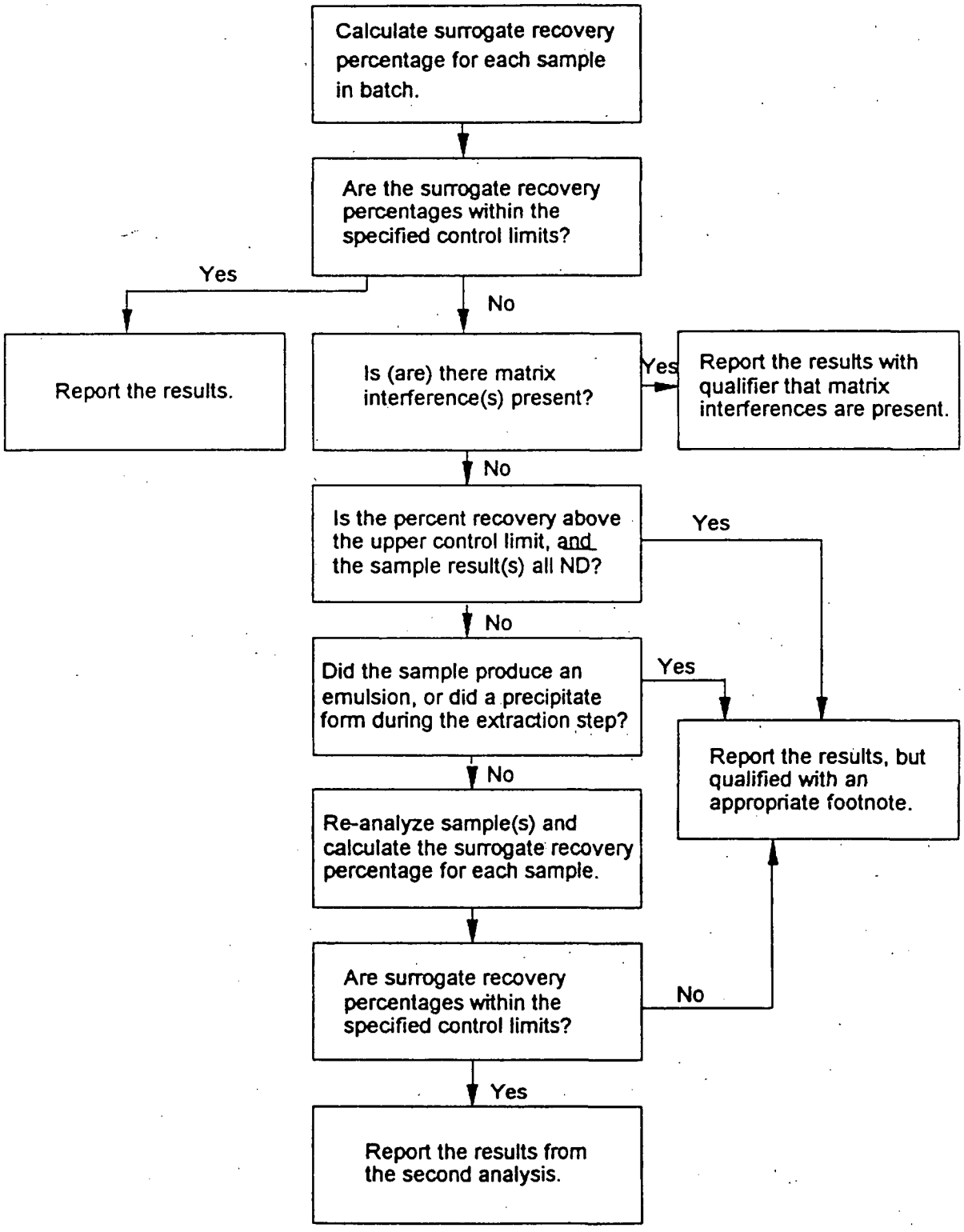


Figure 12-7
Evaluation of Duplicate Sample and/or Duplicate Matrix Spike Results

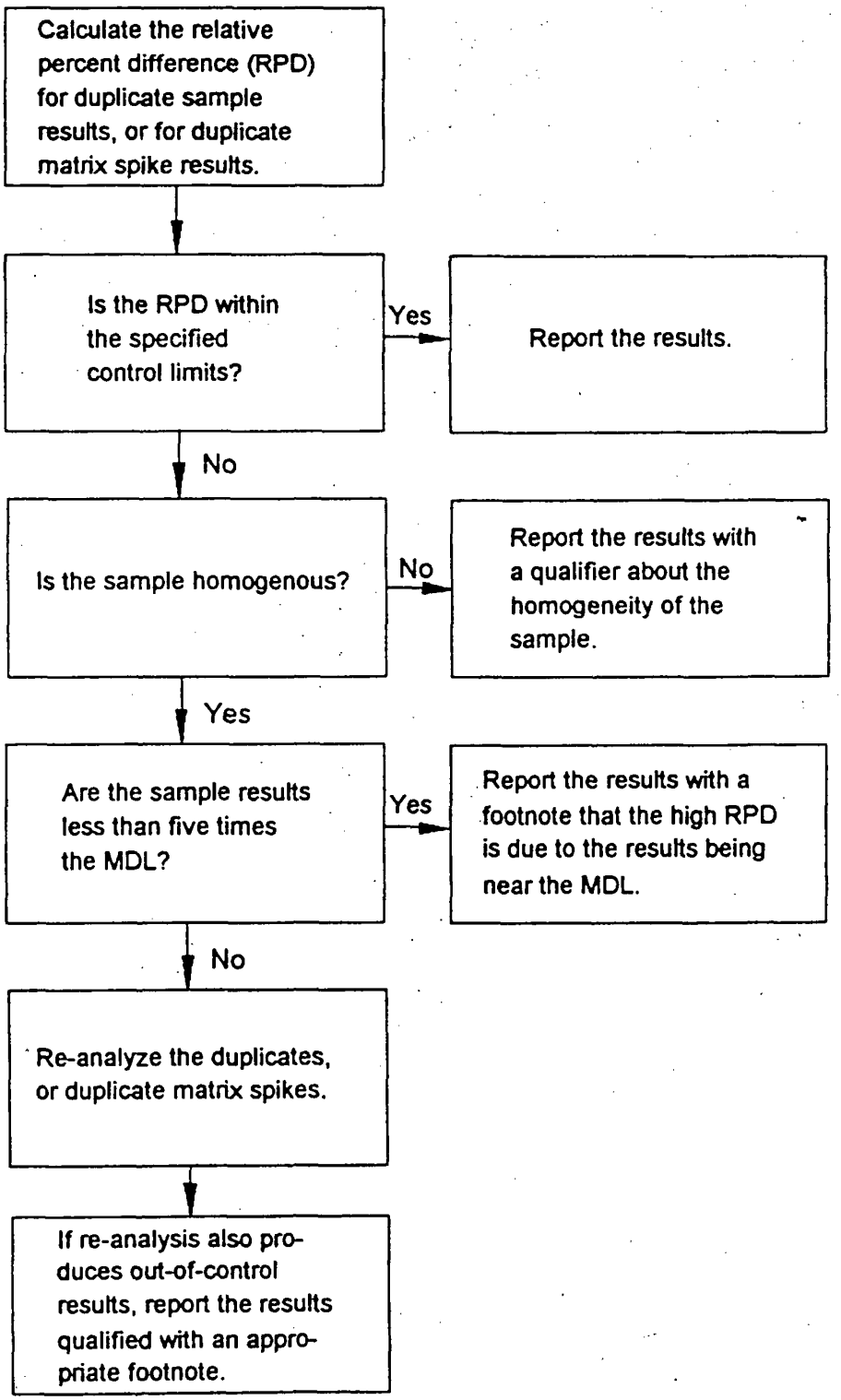


Figure 12-8
Evaluation of Matrix Spike Recoveries

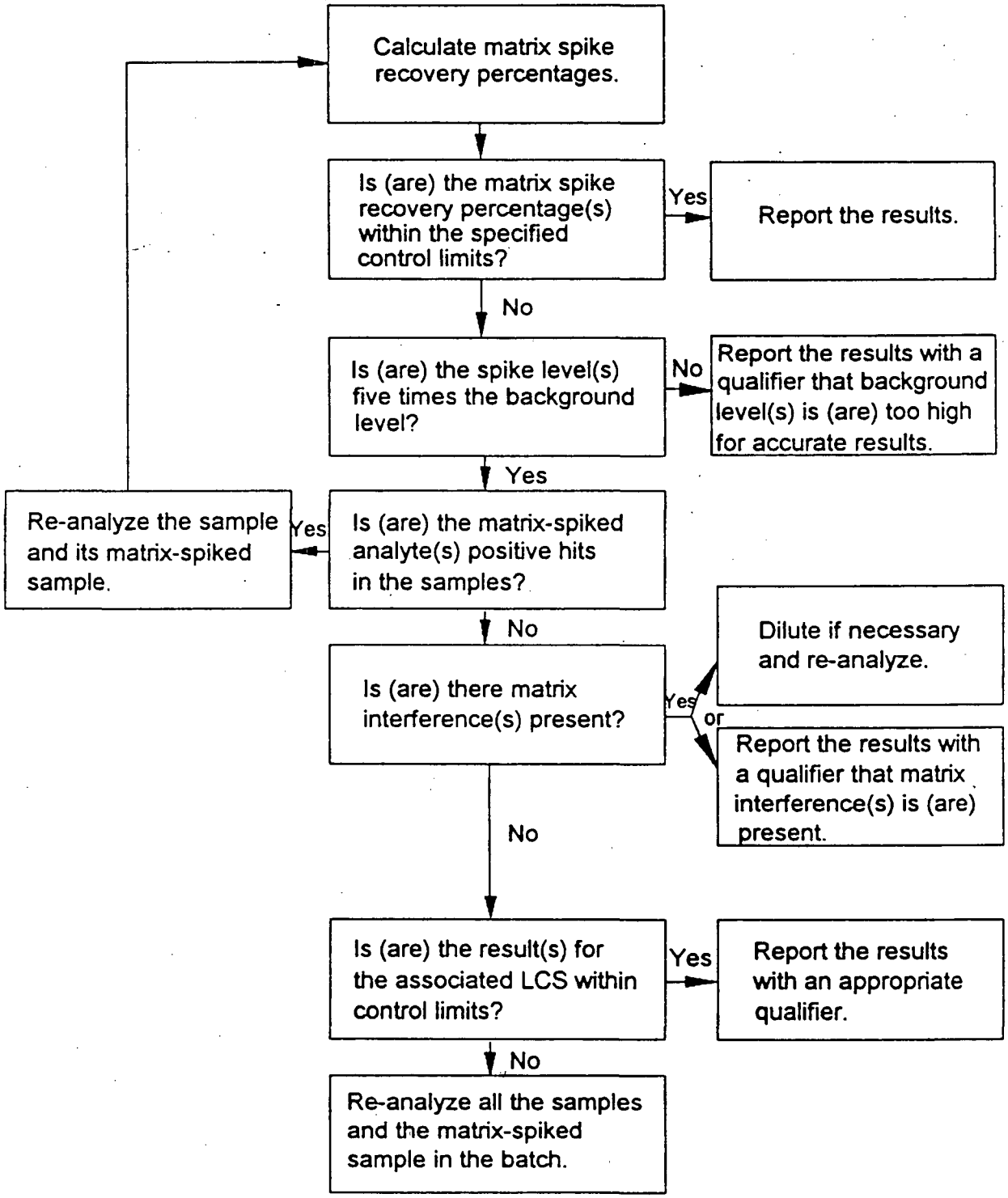


Figure 12-9
Evaluation of Laboratory Control Sample (LCS) Results

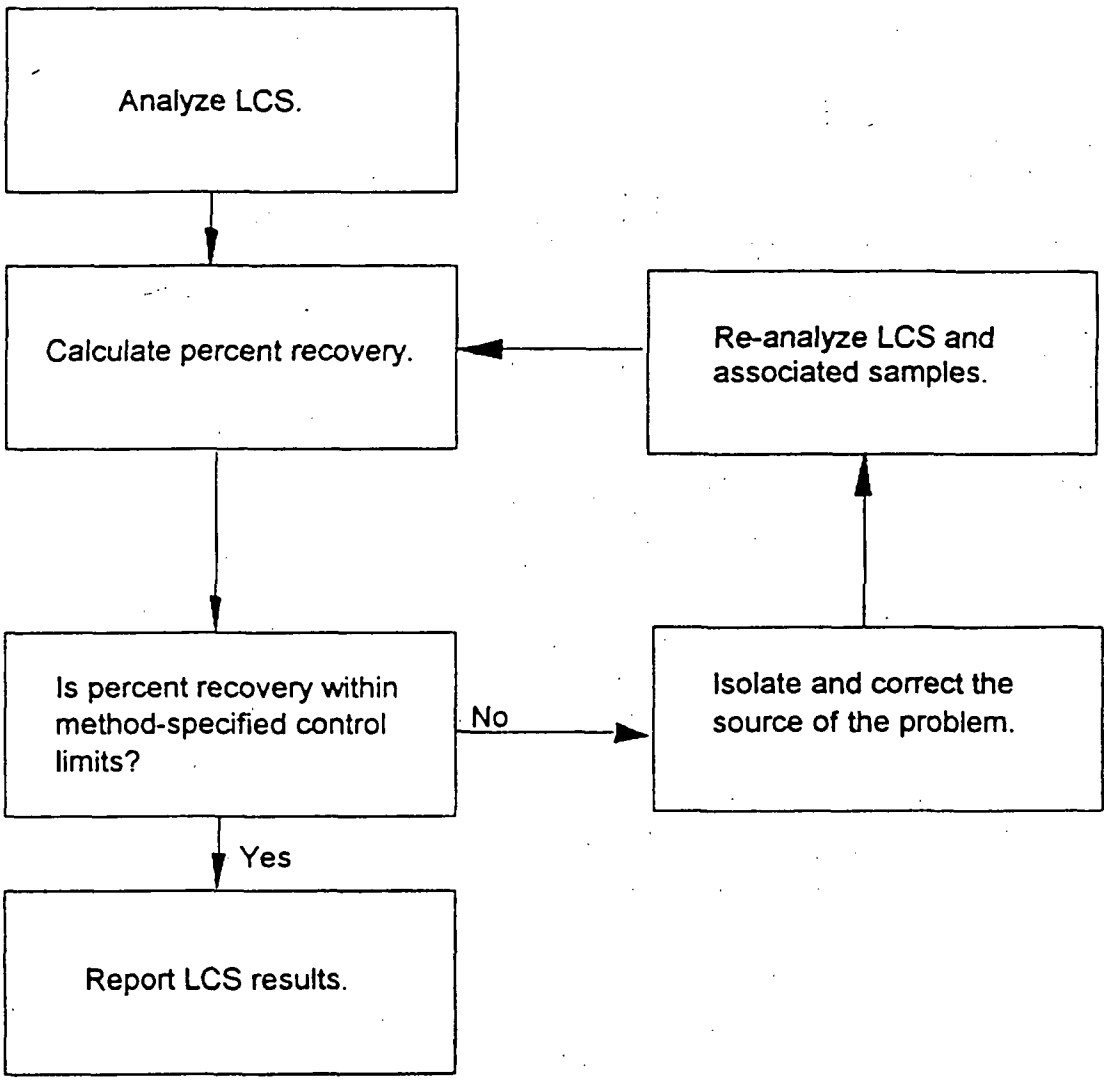


Table 12-1
Descriptions of CAS Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results for appropriate organic methods, including associated EPA or CAS acceptance criteria
5. Chain of custody documents
6. Dates of extraction and analysis for all tests

Tier II. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery, including associated EPA or CAS acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference

Tier ADEC. In addition to the Tier I Deliverables, this CAR includes the following:

1. Laboratory control sample/duplicate laboratory control sample results, with calculated recovery and/or associated acceptance limit criteria
2. Results of initial and continuing calibration verification standards analyses, with calculated recoveries
3. Copies of the raw data for method blank(s) and sample(s)

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or EPA QC check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Mass spectra and summary of TIC compounds will be included upon request.

Tier IV. CLP Data Packages

A complete Contract Laboratory Program (CLP) data package, utilizing CLP methods, CLP forms, and fulfilling all deliverable requirements, as specified in the EPA CLP Statement of Work. The data package may include diskette deliverables, upon request.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of CAS/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing performance evaluation samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Additionally, internal system audits of CAS/Kelso are conducted regularly by the Quality Assurance Manager and by the CAS Quality Assurance Director. The internal system audits are scheduled as five auditing events as follows:

- Comprehensive lab-wide system audit - annually during 1st calendar quarter
- Comprehensive "vertical" project audits examining compliance with all QA program requirements as applied to selected projects - 2 per year
- Focused audits examining the lab-wide implementation of a selected QA program requirement - 2 per year.

The results of each audit are reported to the Laboratory Director for review and comment. Any deficiencies noted by the auditor are summarized in an audit report and corrective action is taken within a specified length of time to correct each deficiency. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification if not already provided.

13.2 Performance Audits

CAS/Kelso also participates in the analysis of performance evaluation (PE) samples. Participation in PE studies is performed on a regular basis and are designed to evaluate all analytical areas of the laboratory. In addition to those PE studies required by programs listed in Table 13-1, CAS participates in additional studies as follows:

- Water Pollution (WP) and Water Supply (WS) PE studies, equivalent to past USEPA studies.
- Environmental Resource Associates (ERA) Soil PE studies, 2 per year.
- ERA Water PE studies, "non-WS,WP" parameters, 2 per year.
- ERA Underground Storage Tank PE studies, 2 per year.
- USEPA Microbiology (WSM) PE studies, 2 per year.

PE samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections prepare an analytical report, which is forwarded to the QA Manager for subsequent reporting to the appropriate agencies or study coordinator. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1
Current CAS Performance and System Audit Programs

Federal and National Programs

- American Association for Laboratory Accreditation (A_2LA)
  Environmental Laboratory Accreditation No. 0490-01
- Naval Facilities Engineering Service Center (formerly NEESA)
  Approved Laboratory for Drinking Water, Wastewater and Hazardous Waste
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE)
  Approved Laboratory for Drinking Water, Wastewater and Hazardous Waste
- U.S. Army Corps of Engineers - MRD, HTRW Mandatory Center of Expertise
  Validated for Drinking Water, Wastewater and Hazardous Waste
- USEPA, Contract Laboratory Program (CLP)
  Contract 68-D5-0135

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory
  Drinking Water Laboratory
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Delaware, Delaware Health and Social Services
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Environmental Water Testing Certification No. E87412
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Massachusetts, Department of Environmental Protection
  Certified Laboratory No. M-WA035
- State of Minnesota, Department of Health
  Certified Environmental Laboratory - effective April 1999.
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Tennessee, Department of Environment and Conservation, Div. of Underground Storage Tanks
  UST Approved Laboratory
- State of Oregon, Department of Human Resources, Health Division
  Certified Drinking Water Laboratory No. WA035
- State of Utah, Department of Health, Division of Laboratory Services
  Certified Environmental Laboratory
- State of Washington, Department of Health
  Certified Drinking Water Laboratory No. 017
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Certification No. C001

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of Columbia Analytical Services Quality Assurance program. Instruments at CAS (e.g., GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on **every** instrument used at CAS before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is **required** before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received.;
- Date the equipment was placed into service.;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase. Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D. This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer.

When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

Failure to meet established analytical controls, such as the quality control objectives outlined in Section 9.0, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

Problems with analysis, as well as the corresponding corrective actions taken, are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the *SOP for Nonconformity and Corrective Action Documentation* (SOP No. ADM - NCAR). This form is utilized to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that effective corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Corrective action due to a performance audit or a check sample problem is initiated by the Quality Assurance Manager; the affected laboratory personnel are promptly informed, as are the laboratory supervisors and managers.

Figure 15-1

COLUMBIA ANALYTICAL SERVICES, INC.

Nonconformity and Corrective Action Report

SAMPLES/SYSTEM/JOB/CLIENT AFFECTED

NONCONFORMITY

Analysis/Event: _____

Instrument/System: _____ Date: _____

Detailed Description:

Originator: _____ Date: _____

Supervisor Verification: _____ Date: _____

CORRECTIVE ACTION AND OUTCOME

Detailed Description: (Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the problem. Describe the outcome.)

Person Responsible: _____ Date: _____

Supervisor Verification: _____ Date: _____

NOTIFICATION - CUSTOMER/CLIENT - INTERNAL/EXTERNAL

Project Chemist Notified? No Yes: Date: _____

Customer Notification Necessary? (Attach telephone record) No Yes: Date: _____

Notifier: _____ Date: _____

ACCEPTANCE OF CORRECTIVE ACTION

QA Coordinator: _____ Date: _____

Original: Client File Photocopies: Supervisor and QA Coordinator CORACTRP.DOC 10/29/96

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Information flow and feedback mechanisms are designed so that analysts, supervisors and managers are aware of quality assurance issues in the laboratory.

Analysts performing routine tests in the laboratory are aware of the various method acceptance criteria and in-house control limits that must be met in order to generate acceptable results. The analysts are also responsible for generating a Data Quality Report (DQR) form with every analytical batch they process; this report contains explicit documentation of the various controls that must be met during the analysis. The DQR form also allows the analyst to provide appropriate notes and/or a case narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. This may or may not supersede the laboratory's own DQR depending on the nature of the problem. Supervisors review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must also accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists if required to do so (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all performance evaluation sample and analysis programs, and for all laboratory certification/accreditation responsibilities.

The QAM also prepares quarterly reports for the Laboratory Director which summarize the various QA/QC activities that have occurred during the previous quarter. The typical report will address such topics as the following:

- Status, schedule, and results of internal and external audits (e.g., deficiency resolution status);
- Status, schedule, and results of internal and external performance evaluation studies;
- Status of certifications, accreditations, and approvals;
- Status of SOPs review;
- Status of MDLs studies;

- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

Any problems noted by the Laboratory Director are then discussed during the regularly-scheduled senior staff operations meetings with all appropriate department managers. The Laboratory Director performs an annual documented review of the quality system.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Information of previously acquired skills and abilities for a new employee is entered into a centralized database (namely, First Resource) maintained by the Human Resources personnel. The database is also used to record the various technical skills and abilities acquired and maintained by an employee while employed by CAS. Information in the database includes the employee's name, a description of the skill including the appropriate method reference, the name of the supervisor who certified completion of the training, and the date the training was completed. Technical training is documented following the *SOP for Documentation of Technical Personnel Training* (SOP No. ADM-TRANDOC).

Training begins the first day of employment at CAS when the company policies are presented and discussed. Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the analysis of quality control samples qualifies the analyst to perform the method independently. An periodic demonstration of proficiency is required to maintain continuing qualification, as described in the *SOP for Documentation of Technical Personnel Training* (SOP No. ADM-TRANDOC).

Safety training begins with the reading of the *Safety Manual*. All employees must pass a safety test within the first month of employment. All employees are also required to attend monthly safety meetings during which the safety program is discussed and safety training is presented by the Environmental, Health and Safety Officer.

CAS encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, laboratory-specific training exercises and performance of external (independent) PE sample analyses are all used to provide employees with professional growth opportunities.

Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all processes at CAS.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), and Proposed Update IVA (January 1998). See Chapters 1, 2, 3, and 4.

- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).

- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).

- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplement I, EPA/600/R-94/111 (May 1994).

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.

- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplement I, EPA/600/4-90/020 (July 1990) and Supplement II, EPA/600/R-92/129 (August 1992) and Supplement III, EPA/600/R-95/131 (August 1995).

- *Standard Methods for the Examination of Water and Wastewater*, 16th Edition (1985); 17th Edition (1989); 18th Edition (1992); 19th Edition (1995). See Introduction in Part 1000.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- 40 CFR Part 141, National Primary Drinking Water Regulations.

- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Massachusetts, Oregon, Washington, Wisconsin, etc.).

- Annual Book of ASTM Standards, Part 31, Water.

- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM01.8, OLM02.0, OLM03.1, and OLM03.2.

- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0.

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).

- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).

- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.

- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).

- *Identification and Listing of Hazardous Waste*, California Code of Regulations, Title 22, Division 4.5, Chapter 11.

- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).

- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).

- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

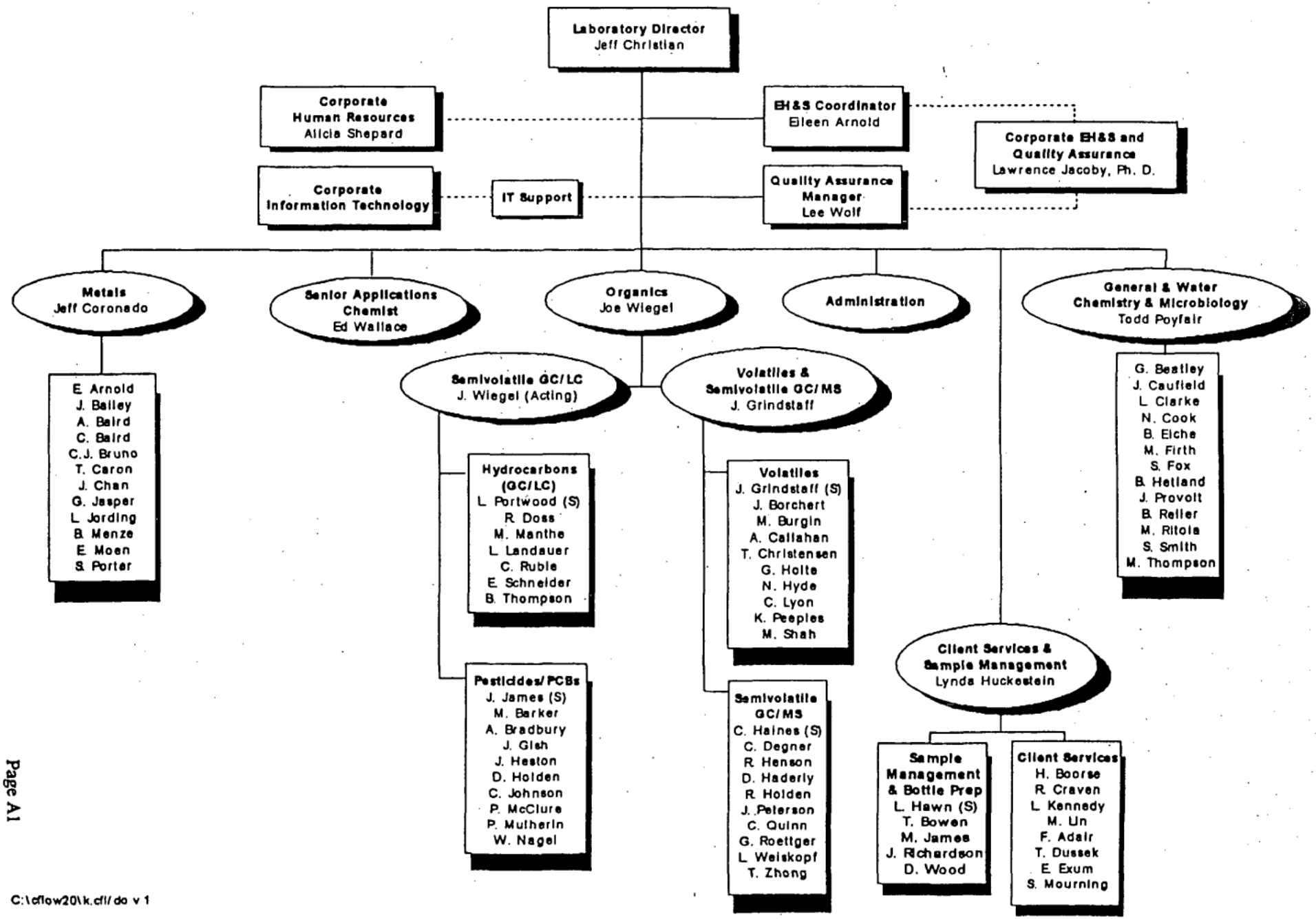
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).

- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).

APPENDIX A

ORGANIZATIONAL CHART and RESUMES OF KEY PERSONNEL

Columbia Analytical Services, Inc.
Kelso, Washington Laboratory Organization



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JEFFREY D. CHRISTIAN AUGUST 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Vice President/Kelso Laboratory Director. January 1996-Present.**

Responsibilities All phases of laboratory operations, including project planning, budgeting, and quality assurance. Also responsible for additional duties aquired as a member of the Columbia Analytical Services, Inc. Board of Directors.

Experience

Kelso Laboratory Director - Columbia Analytical Services, Inc., Kelso, Washington. All phases of laboratory operations, including project planning, budgeting, and quality assurance. 1993-1995.

Kelso Laboratory Operations Manager - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibility was directing the daily operation of the Kelso Laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, Project Manager for State of Alaska Department of Environmental Conservation contract, and Coordinator for EPA Special Analytical Services (SAS). 1992-1993.

Project Chemist and Manager, Metals Analysis Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. 1989-1992.

Scientist - Weyerhaeuser Technology Center, Federal Way, Washington. Supervised atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. 1986-1989.

Lead Technician, Metals Lab - Weyerhaeuser Technology Center, Federal Way, Washington. Primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. 1981-1986.

Research Assistant - ITT Rayonier, Olympic Research Division, Shelton, Washington. Performed water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry. 1978-1981

Education

Evergreen State College, Olympia, Washington. B.S. Chemistry. (b) (6)
VG-Elemental ICP-MS Training Course (b)
Pacific Lutheran University, Tacoma, Washington. (b) (6)
Tacoma Community College, Tacoma, Washington (b) (6)
Perkin-Elmer Advanced Furnace, Norwalk, Connecticut. (b)
L.H. Bates Technical, Tacoma, Washington - Chemistry Certification. (b) (6)
Central Washington University, Ellensburg, Washington (b) (6)

Publications/ Presentations

ICP-MS Analysis of Marine Tissue, AOAC Meeting, Olympia, Washington, June 1994.

Analyzing Seawater by ICP-MS, Environmental Lab, October/November 1993, Vol. 5. No. 5, page 10-13.

Reductive Precipitation as a Pre-Concentration Technique for the Analysis of Seawater by ICP-MS, June 1993, AOAC, Olympia, Washington.

The Analysis for Metals at PPT Levels in Environmental Samples Using Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Techniques, HazMat West, November, 1992, Long Beach, California.

Analysis of Pulping Liquor by ICP Emission, January, 1984. International Conference on Plasma Spectrochemistry, San Diego, California. (Co-Author).

Modification of ICP for Sulfur Determination in Black Liquors, January, 1984. International Conference on Plasma Spectrochemistry, San Diego, California. (Co-Author)

LEE E. WOLF *OCTOBER 1988-PRESENT*

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position Responsibilities

Kelso Quality Assurance Manager. 1996-Present.

Responsible for the overall coordination of the laboratory QA program and for ensuring that established quality objectives are met. Responsible for Quality Assurance functions including the Quality Assurance Manual, certifications, documenting standard operating procedures, and maintaining performance evaluation records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits. Provides audit responses and initiates any changes in procedures resulting from an audit. Coordinate the analysis of performance evaluation samples required for certification/accreditation programs. Report and review results for these analyses. Conduct internal audits and make recommendations for corrective action.

Experience

Project Chemist/Principal Organic Scientist - Columbia Analytical Services, Inc., Kelso, Washington. Perform GC and GC/MS method development, and special projects coordination. Oversees and acts as technical advisor to the GC and GC/MS laboratories. Acts as GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions including managing and coordinating projects for clients, identifying client's needs, and preparing data reports. 1994-1996.

Semivolatile Organics Department Manager - Columbia Analytical Services, Inc., Kelso, Washington. Overall management of the semivolatile organics analysis section. Oversee the operation of semivolatile GC/MS instrumentation, data review and reporting, and related QA/QC functions. Responsible for the supervision of staff, including scheduling, training, and other personnel issues. Also responsible for Project Chemist functions for organics EPA-SAS and other clients. Involved coordinating and scheduling projects for clients, identifying client's needs, and preparing data reports (1992-1994). 1988-1994.

GC/MS Chemist - U.S. Testing Co., Richland, Washington. Responsible for GC and GC/MS analysis of water and soil samples for volatiles and semivolatiles by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Experience also with pesticide/PCB analysis by EPA Methods 8080 and CLP. Responsible for development of analysis methods for non-routine pesticides and herbicides and performed HPLC analysis. 1985-1988.

Laboratory Assistant - Eastern Washington University, Cheney, Washington. Responsible for supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety. 1985.

Chemist Assistant - Spokane County Air Pollution Control Authority, Spokane, Washington. Responsible for gathering and analyzing air samples for CO content using IR equipment. 1984.

Education

Documenting Your Quality System, A2LA Short Course, Las Vegas, Nevada (b)
Internal Laboratory Audits, A2LA Short Course, Las Vegas, Nevada (b)
Mass Spectra Interpretation, ACS Short Course, Denver, Colorado (b)
Eastern Washington University, Cheney, Washington - B.S. in Chemistry. (b)
Eastern Washington University, Cheney, Washington - B.A.(minor) in Geology (b)

Publications/ Presentations

Selected Ion Monitoring: Issues for Method Development, Panel Discussion, Association of Official Analytical Chemists, (AOAC) Pacific Northwest Regional Meeting, 1995.
Method Enhancement Techniques for Achieving Lowlevel Detection of Butyl Tin in Marine Sediments and Tissues, Association of Official Analytical Chemists (AOAC Pacific Northwest Regional Meeting, 1994.
The Determination of Low-Level Concentrations of Polynuclear Aromatic Hydrocarbons (PAHs) in Soil and Water Using Gas Chromatography/Mass Spectroscopy Selected Ion Monitoring (GC/MS SIM), HazMat West, November, 1992, Long Beach, California.

Memberships

American Society for Mass Spectrometry, 1995-present.
American Chemical Society, 1989 to present.

EILEEN M. ARNOLD DECEMBER 1987-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Kelso Laboratory Health and Safety Officer, Scientist IV, Metals Laboratory. 1987-Present.

Responsibilities

Kelso Laboratory Health and Safety Officer: Responsible for the development and implementation of the CAS Health and Safety Program. Duties include accident investigation and incident review, and maintenance of all safety-related equipment and documents. Responsible for supervising the Laboratory Waste Management Plan. Chemical Hygiene Officer. 1987-Present.

Scientist IV, Metals Laboratory: Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. 1987- 1992 and 1994-Present.

Experience

Project Chemist, Client Services Group - Columbia Analytical Services, Inc., Kelso, Washington. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. 1992- 1994.

Chemist - Dow Corning Corporation, Springfield, Oregon. ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. 1986-1987.

Chemist - Ametek, Inc., Harleysville, Pennsylvania. Product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. 1982-1985.

Chemist - Janbridge, Inc., Philadelphia, Pennsylvania. Maintained electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards. 1978-1982.

Education

Immaculata College, Immaculata, Pennsylvania - B.A. in Chemistry. (b)

Affiliations

American Chemical Society. 1987.

LYNDA A. HUCKESTEIN JANUARY 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position Client Services Manager

Responsibilities Senior Project Chemist/Management of Client Services. Includes responsibilities for Project Management, Marketing, Report Production and Sample Management. 1998-Present.

Experience

Senior Project Chemist- Responsible for approximately $1.5 million dollars of client work annually, Lynda's primary responsibilities include technical project management and client service. She is responsible for meeting her clients' needs and providing appropriate analysis and timely reporting. Her area of expertise includes pulp & paper, marine, mining and DOD project management. 1992-1998

Report Generation Supervisor- Lynda also leads a group of five report generation staff responsible for the generation of both the hardcopy report deliverables and the electronic data deliverables. Her role is to manage the coordination of client specifications with the current laboratory data reporting platform. She also facilitates continuous quality improvement projects within the report production group.

Project Chemist, Fort James - For over three years Lynda has been the primary liaison between the Fort James facilities in Camas, Washington; Wauna and Portland Oregon; and Kalamazoo, Michigan. Columbia provides analytical testing and consultation in support of Fort James' NPDES analyses, waste stream identification and FDA paper testing.

Project Chemist, Rayonier - Columbia has been working with Rayonier's Port Angeles facility for three years. Lynda is currently providing project oversight on monthly AOX analyses by EPA Method 1650, NPDES monitoring and with analytical work pertaining to a sensitive site investigation.

Project Chemist, Cominco Alaska, Inc. - Lynda has been project manager for Cominco work at their Red Dog Mine in Alaska for over eight years. She provides technical and regulatory interpretation assistance as well as project organization to their work sent to the laboratory. Cominco's NPDES testing requires a quick result turn-around-time on a large volume of samples, often within 24-72 hours of sample receipt.

Project Chemist and Department Manager, General and Water Chemistry and Biology Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included the management of General and Water Chemistry, and the Biology Laboratory, which consisted of routine wastewater, bioassay, and microbiological analyses, supervision, data review, and reporting. 1989-1992.

Laboratory Analyst III - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and Total Kjeldahl Nitrogen (TKN), in addition to miscellaneous wet chemistry. 1989.

Microbiologist/Chemist - Coffey Laboratories, Portland, Oregon. Coliform analysis; water chemistry. 1983.

Laboratory Assistant - Oregon State University, Corvallis, Oregon. Wheat spike dissection and tissue culture. 1983.

Education Oregon State University, Corvallis, Oregon - B.S. in Microbiology. (b)

Affiliations Chairperson for the City of Longview Solid Waste Recycling/Composting Committee, (b)

JEFFREY A. CORONADO APRIL 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Metals Department Manager. October 1992-Present.

Responsibilities

Management Duties: Responsibilities include management of all aspects of the metals laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts.

Technical Duties: Primary analytical responsibility is trace level metals analysis by ICP-MS. Analysis range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Currently developing methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.

Experience

Supervisor, GFAA Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation. April 1989-October 1992.

Education

EnSys Inc. Field Immunoassay Training Course (b) (6)

Winter Conference on Plasma Spectrochemistry, San Diego, CA (b)

VG-Elemental ICP-MS Training Course. 1992.

Western Washington University, Bellingham, Washington - B.S. in Chemistry. (b)
Western Washington University, Bellingham, Washington - B.A. in Business Administration. (b)

GREGORY P. JASPER JUNE 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Scientist IV, Supervisor, Metals Digestion Laboratory. 1994-Present.**

Responsibilities Primary responsibilities include designating work assignments to technicians and reviewing technicians' data, training new employees, writing final reports, and quality control. Serves as primary analyst on the inductively coupled plasma mass spectrometer (ICP/MS).

Experience

Senior Analyst, Supervisor, Metals Digestion Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities include designating work assignments to technicians and reviewing technicians' data, training new employees, writing final reports, and quality control. Serves as primary analyst on the inductively coupled plasma mass spectrometer (ICP/MS). 1992-1994

Analyst III, Supervisor, Metals Digestion Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included supervision of Digestion Laboratory personnel. Organization and assignment of work for Digestion Laboratory. Perform Mercury Analysis (lead person) and Flame Atomic Absorption. 1991-1992

Analyst II, Supervisor, Metals Digestion Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included supervision of Digestion Laboratory personnel. Organization and assignment of work for Digestion Laboratory. Perform Mercury Analysis (lead person) and Flame Atomic Absorption. 1990-1991

Analyst I, Metals Analysis Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included sample preparation and digestion for metallic constituents; mercury analysis (lead person); flame atomic absorption, and back-up graphite furnace operation. 1989-1990.

Chemical Technician - James River Technical Service Group, Camas, Washington. Performed analysis of pulping liquors. 1988 -1989.

Education

Winter Conference on Plasma Spectrochemistry, Fort Lauderdale, FL (b)
Clark College, Vancouver, Washington - A.A. in Chemical Technology. (b)

TODD POYFAIR AUGUST 1991-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

General Chemistry Department Supervisor/Manager. 1995-Present.

Responsibilities

Primary responsibilities include the management, supervision, training, workload coordination, data review, reporting, and maintenance of the General Chemistry Laboratory.

Experience

Project Chemist, Client Services Group - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within CAS. 1993-1995

Scientist II, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities include the review and summarization of pH, alkalinity, conductivity, turbidity, hardness, and CODs. 1992-1993

Scientist I, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for analysis of Total Organic Halogens, Chemical Oxygen Demand, Sulfides, Ammonia, Total Kjeldahl Nitrogen, Nitrate/Nitrite by Lachat, and Cyanide. 1992

Analyst III, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for the analysis of pH, Conductivity, Alkalinity, Turbidity, and Oil and Grease. 1991-1992

Education

Portland State University, Portland, Oregon - B.S. in Chemistry (b) (6)
Portland State University, Portland, Oregon B.A. in German. (b)
Brigham Young University, Provo, Utah. (b) (6)

JOSEPH V. WIEGEL OCTOBER 1992-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Organics Laboratory Manager 1998-Present.

Responsibilities

Oversees the operation of the organic groups which consists of four separate laboratories: GC/MS Semivolatiles Laboratory, GC Pesticide Laboratory, Petroleum Hydrocarbon Laboratory, and the Volatiles Laboratory. Responsible for the quality and timeliness of the organic laboratory's analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

Experience

Business Development Manager - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for marketing and business development activities for Washington, Oregon, Idaho and other areas in the Northwest as designated. These activities include: development of marketing and sales plans and business development strategies, initial prospective client contact, proposal and SOQ development, trade show participation, arranging for the delivery of technical presentations, and other duties as identified. 1996-1998

Supervisor/Project Chemist, Petroleum Hydrocarbon Group - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for management, training, workload coordination, data review and reporting, instrument operation and maintenance, and method development of the petroleum hydrocarbons department. Responsibilities also included interfacing with clients to provide technical project management and customer service. As Project Chemist, duties included working with clients to provide timely, appropriate, and quality analytical services, coordinating with CAS laboratory and administration to ensure that analyses are properly executed and meet the clients' needs, and acting as liaison for all client-related activities within CAS. 1994-1996

Project Chemist/Client Services Group - Columbia Analytical Services, Inc., Kelso, Washington. Responsibilities included interfacing with clients to provide technical project management and customer service. Duties included working with clients to provide timely, appropriate, and quality analytical services, coordinating with CAS laboratory and administration to ensure that analyses are properly executed and meet the clients' needs, and acting as liaison for all client-related activities within CAS. 1992-1994

Organic Extractions Laboratory Manager - Versar Laboratory, Inc., Springfield, Virginia. Responsible for directing the activities of the organic extractions laboratory, which included sample preparation, sample extraction, extract cleanup, and extract screening prior to analysis by GC and GC/MS. 1989-1992.

Education

Environmental Issues in Mineral Exploration, NWMA Short Course, (b)
Contaminated Sediments, Environmental Law Training Center (b)
Basic Gas Chromatography, Restek Corp. (b) (6)
Georgetown University, Washington, D.C. - B.S. in Biology. (b)

Memberships

Association of Official Analytical Chemists, 1998-Present
Society of Environmental Toxicology and Chemistry, 1998-Present
Associated Oregon Industries, 1997-Present
Northwest Environmental Business Council, 1997-Present
Northwest Mining Association, 1996-Present

JEFF A. GRINDSTAFF OCTOBER 1991-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Manager, GC/MS Volatile and Semivolatile Organics Laboratory. 1997-Present.

Responsibilities

Primary responsibilities include supervising GC/MS volatile and semivolatile organics staff, new method development, training, reviewing GC/MS volatile organics data, tracking department workload, scheduling and performance of volatile organics analyses, and general maintenance and troubleshooting of GC/MS systems.

Experience

Manger, GC/MS Volatile Organics Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities include supervising GC/MS volatile organics staff, new method development, training, reviewing GC/MS volatile organics data, tracking department workload, scheduling and performance of volatile organics analyses, and general maintenance and troubleshooting of GC/MS systems. 1994-1997.

Scientist III, GC/MS Volatile Organics Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. 1991-1994

Chemist - Enseco-CRL, Ventura California. Responsible for establishing GC/MS department, including maintenance of consumable inventory; preparing a state certification data package; and for developing and implementing analytical methods, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and nonroutine volatile organic analyses to meet holding times and due dates. 1990-1991.

GC/MS Chemist - Coast-to-Coast Analytical Services, San Luis Obispo, California. Prepared daily analytical standards for volatile organic analyses, and performed daily calibration and tuning of GC/MS instrumentation, along with general maintenance of hardware. Implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants. 1988-1990.

Education

Mass Selective Detector Maintenance, Hewlett-Packard Education Center. (b) (6)
Interpretation of Mass Spectra I, Hewlett-Packard Analytical Education Center. (b) (6)
California Polytechnic State University, San Luis Obispo, California - BS in Chemistry. (b)
Allan Hancock College, Santa Maria, California - AA in Liberal Arts. (b)

Publications/ Presentations

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, Florida. 1996.

Affiliations

American Chemical Society. (b)

1VID L. EDELMAN APRIL 1988-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Vice President-CAS, Corporate Technical Director, Information Technology Director, and East Coast Laboratories Director** - 1992 to present.

Responsibilities Company Operations Committee Member, Member of Board of Directors. Technical Director and Senior Project Manager. Responsible for implementing new technology, TQM, and laboratory mergers. Responsible for the all operations in the Southeastern United States.

Experience **Laboratory Director** - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for the overall operation of the Kelso laboratory. 1991-1992.

Organics Laboratory Manager - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for operating/managing the organic and bioassay laboratories to produce high quality, cost-effective, timely analytical services to comply with Federal RCRA, NPDES, CERCLA, SDWA, UST and State Regulatory requirements. 1988-1991.

Laboratory Manager - James River Corporation, Environmental Services. Responsible for managing laboratory operations, using GC/MS/DS, ICP, AA, TOC, GC/FID/ECD/TCD, UV/VIS, and bioassay techniques on effluents, sludges, wastes, ground water, and process streams. Responsible for obtaining accreditations from three state regulatory agencies. 1986-1988.

Laboratory Coordinator - Crown Zellerbach Corporation, Environmental Services. Performed/supervised analytical sampling services for RCRA Remedial Investigation/Feasibility Studies, NPDES permits, ground water contamination studies, and solid waste disposal site investigations. Performed GC/MS, HPLC, ICP, and wet chemistry techniques. Developed specialized skills for analyzing materials from pulp/paper/packing industry. 1979-1986.

cation University of Washington, Seattle, Washington - Ph.D. in Forest Resources. (b) (6)
University of Washington, Seattle, Washington - M.S. in Organic Chemistry.
University of Washington, Seattle, Washington - B.S. in Chemistry. (b)

Publications/ Presentations

Cost-Effective Dioxin Characterization using the P450 Reporter Gene System (RGS), with Bob Wilkinson, Mark Butler, and Jennifer Jones. 4th Annual Meeting of NAC Society of Environmental Toxicology and Chemistry, May 1998, Saratoga Springs, NY.

Cluster Rule Method Flexibility, Contracting for Analytical Data -Contract Lab Perspective, NCASI, Southern Regional Meeting, April 1997 and NCASI, Western Regional Meeting September, 1997.

Utilization of Lignin Degradation Compounds to Determine Groundwater Contamination from a Pulp and Paper Mill Landfill, with Daniel Diehl, Jeff Pickrell, and Craig Myers. TAPPI National Meeting, Minneapolis/St. Paul, Minnesota, 1997.

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with Kairas Parvez, Jeff Grindstaff, and Paul Laymon. TAPPI National Meeting, Orlando, Florida, 1996.

P450 RGS: A Biomarker for Assessing the Toxicity of Environmental Samples, with Jack Anderson, Kristen Bothner, and Steve Vincent. TAPPI National Meeting, Atlanta, Georgia, 1995.

*Investigation of Hydrocarbons Detected Downgradient from a Woodwaste Disposal Site.*with Kirk Girard, TAPPI National Meeting, Portland, Oregon 1994.

Hazardous Waste Analyses, The Pollution Prevention Network, 01/27/93.

Proposed Acute Whole Effluent Toxicity Test, AWB Stormwater Permitting Seminar, Seattle, Washington. 1992.

Analytical Considerations for NPDES Permitting, 56th Annual PNPCA Conference, Eugene, Oregon. 1989.

Organic Analysis Quality Assurance, PNWS-AWWA Conference, Eugene Oregon. 1989.

Experience Performing Environmental Analysis of the Pulp and Paper Industry, ACS Northwest Section Meeting, Portland, Oregon. 1989.

EPA Method 200.7 for Trace Metals Analysis, AOAC National Meeting, Seattle, Washington. 1986.

Chemical Analysis of Wastes for RCRA Compliance, Mt. Hood Community College Seminar, Portland, Oregon. 1986.

Drinking Water Certification Using ICAP Techniques, AOAC Regional Meeting, Olympia, Washington. 1984.

Affiliations American Chemical Society. 1976-Present. *Standard Methods* Committees Participation
Technical Association of the Pulp and Paper Industry. 1972-Present
Association of Official Analytical Chemists. 1979-Present.

LAWRENCE J. JACOBY AUGUST 1990-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Quality Assurance Director and Environmental, Health and Safety Director, Vice President. 1992-Present.

Responsibilities

Responsible for the conduct of the quality assurance (QA) and environmental, health and safety (EH&S) programs and activities for CAS; for conducting QA and EH&S audits at each CAS laboratory to ensure that the QA and EH&S objectives established by management and by the various certifications, accreditations, project plans, and regulations under which CAS operates are satisfactorily met, to ensure that the QA and EH&S programs are functioning as stated in QA Manuals and EH&S Manuals, and to make appropriate recommendations for corrective actions and improvements; for management of performance evaluation and round-robin samples analyses programs; for evaluating data quality; for helping to provide training in support of the CAS total quality program; and for providing technical assistance to project chemists.

Experience

Quality Assurance Coordinator - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for CAS/Kelso's quality assurance program and projects, and for evaluating data quality. 1990-1992.

Client Services Manager - CH_2M Hill Laboratory, Redding, California. Management of client services and sample custody groups; customer service and maintenance; project management; proposal and quotation preparation; project-engineer/laboratory liaison. 1989-1990.

Inorganic Division Manager - CH_2M Hill Laboratory, Redding, California. Responsible for managing the operation of the inorganic analyses section including wet chemical, soil, and metals analyses; project management; customer service; proposal and quotation preparation. 1988-1989.

Laboratory Manager - CH_2M Hill Laboratory, Corvallis, Oregon. Responsible for managing the operation of the laboratory and coordinating the activities of project support staff; project management; quality assurance; proposal and quotation preparation; laboratory safety; engineering project consulting. 1986-1988.

Analytical Chemist - Teledyne Wah Chang, Albany, Oregon. Responsibilities included methods development; instrument maintenance; non-routine analyses; workload scheduling and coordination; and task force assignments. 1976-1986.

Assistant Professor/Instructor - Portland State University, Portland, Oregon. 1969-1971. Chemeketa Community College, Salem, Oregon. 1971-1976. Taught college courses in general, organic and analytical chemistry.

Education

Colorado State University, Ft. Collins, Colorado - Ph.D. in Organic Chemistry. (b)
Oregon State University, Corvallis, Oregon - B.S. in Chemistry. (b)

Affiliations

American Society for Quality Control.
American Chemical Society.
AOAC International.

STEPHEN W. VINCENT JULY 1986-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

President, CAS Laboratories. 1986-Present.

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager - Weyerhaeuser Company, Federal Way, Washington. 1979-1986. Responsibilities: Involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately $2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist - Weyerhaeuser Company, Longview, Washington. 1975-1979. Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Stanford University - Executives Program. Market Strategy for Technology-Based Companies. (b)
University of California at Los Angeles - Department of Business, Engineering and Management. Advanced Technical Management Program. (b)
University of Washington, Seattle, Washington - Completion of course work for MS Pulp and Paper Technology. (b)
University of California at Los Angeles - Graduate School of Engineering and Applied Science. Graduate School of Management. Engineering and Management Program. (b)
University of Washington, Seattle, Washington - B.S. in Oceanography. (b)

Publications/ Presentations

The State of the Environmental Testing Industry, ACIL Western Division 1995 Spring Meeting, Port Ludlow, Washington
Session Chairman, *New Low-Level Procedures and Field Techniques for the Assessment of Chemical Pollutants in the Environment*, HazMat West, November, 1992, Long Beach, California.

Changing Requirements for Chemical and Biological Analysis of Point Source Discharges, EMCON Geology Conference, Stanford University, Palo Alto, California. August 1989.

Laboratory Certification in the Northwest, Annual Conference, American Water Works Association, Eugene, Oregon. May 1989.

Basic Laboratory Skills, NCASI Central Lakes Meeting, Chicago, Illinois. 1982.

Weyerhaeuser Company's Corporate Quality Assurance Program, NCASI, New Orleans, Louisiana. June 1981.

The Impact of Pulp and Paper Effluents on the Water Quality of the Lower Columbia River, with W. G. Hines and S. R. Young. TAPPI Environmental Conference, New Orleans, Louisiana. April 1981.

Weyerhaeuser Company's Effluent Monitoring Program for Toxic Metals, National Council for Air and Stream Improvement, Portland, Oregon. 1977.

Affiliations

American Chemical Society.
Technical Association of the Pulp and Paper Industry.

APPENDIX B

MAJOR ANALYTICAL EQUIPMENT

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY SAMPLE PREPARATION			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Digestion Systems (4): COD (2) Kjeldahl, Labonco 25-place (1)	 1987, 1989 1997	 LM LM	 7 7
Shatter Box - GP 1000	1989	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	3

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (5): Precisa 180A Mettler PM 480 (2) Precisa 240A Mettler BB 3000	 1988 1988,1989 1989 1990	 MM MM MM MM	 11 11 11 11
Autoclave - Market Forge Sterilmatic	1988	LM	6
Calorimeter - Parr 1241 EA Adiabatic	1987	LM	3
Centrifuge - Damon/IEC Model K	1992	LM	11
Colony Counter - Quebec Darkfield	1988	LM	3
Conductivity Meters (2): Amber Science Model 604 YSI Model 34	 1987 1991	 LM LM	 8 8
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	8
Distillation apparatus (Midi) - Easy Still	1996	LM	4
Drying Ovens (5): Shel-Lab Model 1350 F Shel-Lab Model 1370 F VWR Model 1370 F VWR Model 1500E (2)	 1988 1989 1990 1991	 LM LM LM LM	 11 11 11 11
Flash Point Testers (2): Precision Scientific Model 74537 Pensky-Martens Tester ERDCO Setaflash Tester (2)	 1987 1988, 1991	 LM LM	 2 4
Flow-Injection Analyzer - Lachat Quick Chem AE	1990	LM	7
Hydraulic Press (core sample removal)	1991	LM	7

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Ion Chromatographs (3)			
Dionex 2000i with Peaknet Data Systems (2)	1988	LM	7
Dionex DX-120 with Peaknet Data System	1998	LM	7
Ion Selective Electrode Meters (4)			
Fisher Scientific Accument Model 50	1997	LM	9
Fisher Scientific Accument Model 25	1993	LM	9
Orion Model 920A	1990	LM	9
Corning pH/ion Meter Model 135	1992	LM	9
Microscopes (3):			
Olympus BH-2	1987	LM	11
Bausch & Lomb	1988	LM	11
Swift	1988	LM	11
Muffle Furnaces- Sybron Thermolyne Model F-A1730	1991	LM	11
pH Meters (3):			
Beckman 34 (2)	1989	LM	9
Fisher Scientific Accument Model 20	1993	LM	9
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	11
WS Tyler - RX 86	1991	LM	11
Temperature Control - UE 650	1987	LM	11
Total Organic Carbon (TOC) Analyzers (3)			
O-I Corporation, Model 700 (2)	1986, 1993	LM	3
Coulemetrics Model 5012	1997	LM	3
Total Organic Halogen (TOX) Analyzers (2):			
Mitsubishi (MCI) TOX-10	1986	LM	4
Mitsubishi TOX-Sigma	1995	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	5
UV-Visible Spectrophotometers (2):			
Hitachi 100-40 Single Beam	1986	LM	8
Milton Roy 1001 Plus	1991	LM	8
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	11
Busch R-5 Series Single Stage	1991	LM	11
Water Baths/Incubators (5):			
Hach Model 15320 Incubator	1986	LM	6
Precision Model L-6 (2)	1989, 1990	LM	6
VWR 1540	1991	LM	6
Fisher 11-680-626M Incubator	1992	LM	6

MAJOR ANALYTICAL EQUIPMENT

METALS LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (2)			
Mettler AE 200 (1)	1990	MM	13
Mettler BB240 (1)	1988	MM	13
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA 20 plus AA with Graphite Furnace and Flame Systems	1988	LM	4
Varian SpectrAA 300 Zeeman AA and IBM Data Stations (3)	1989	LM	5
Varian SpectrAA 20 with Flame, Cold Vapor, and Hydride Systems	1988	LM	5
Atomic Fluorescence Spectrophotometer - Brook-Rand Model III	1996	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	13
Drying Oven - VWR Model 1370F	1990	LM	13
Extractors (2):			
RCRA EP Toxicity	1986	LM	6
TCLP Extractor	1989	LM	6
Freeze Dryers (2) - Labconco	1988, 1992	LM	5
Muffle Furnace - Thermolyne Furnatrol Model 53600	1991	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	13

ICP LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES): Thermo Jarrell Ash, Model 61E Simultaneous Emission Spectrometer with 32 Analytical Channels	1988	LM	4
Inductively Coupled Plasma Mass Spectrometer (ICP-MS): VG PQ-S	1997	MM	3
Inductively Coupled Plasma Mass Spectrometer (ICP-MS): VG Model Plasma Quad PQ2 Turbo	1991	MM	3

MAJOR ANALYTICAL EQUIPMENT

GC SEMIVOLATILE ORGANICS SAMPLE PREPARATION			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	4
Analytical Balance - Mettler BB240	1987	MM	17
Aspirator pumps - Labconco Cole Parmer (2)	1994	LM	17
Centrifuges (2):			
Adams Model DYNAC	1986	LM	17
Sorvall Model GLC-1	1988	LM	17
Drying Oven - Fisher Model 655 G	1991	LM	17
Evaporators (10):			
Organomation N-Evap (4)	1989,1990(2),1998	LM	17
Organomation S-Evap (4)	1989-1991	LM	17
Rotary - Labconco Cole Parmer (2)	1994	LM	10
Extractors (80):			
Lab-Line Multi-Unit Soxhlet Extraction Heaters (78)	1987-1992	LM	17
Sonifier (Sonic Horn) (3):			
Tekmar (2)	1994	LM	17
Fisher Model 550	1991	LM	17
Freon Distillation Apparatus (22 liter)	1991	LM	5
GPC-Zymark Benchmate GPC - dual column	1993	LM	4
ABC GPC - single column	1998	LM	4
Muffle Furnace - Parflow MIC 6000	1994	LM	17
Sonic Water Bath (3):			
Branson Model 8200 (2)	1991,1992	LM	17
Branson Model 3200	1987	LM	17
Vacuum Pump - Edwards	1992	LM	17

MAJOR ANALYTICAL EQUIPMENT

GC SEMIVOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4):			
Mettler AT 250	1989	MM	17
Mettler BB 300 (2)	1991	MM	17
Mettler BB240	1994	MM	17
Chromatography Data Systems (13)			
HP Enviroquant (12)	1994	LM	10
Waters Maxima (1)	1991	LM	3
Gas Chromatographs (11):			
Hewlett-Packard 5790 GC with HP 7673 Autosampler and ECD Detector	1988	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FPD/NPD Detectors	1989	LM	3
Hewlett-Packard 5890 GC with HP 7673 (7) Autosampler and Dual ECD Detectors (10)	1990, 1991, 1992, 1993, 1995	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FPD/ECD Detectors	1991	LM	4
Hewlett-Packard 5890 GC with HP 7673 (3) Autosampler and Dual FID Detectors	1989,1991,1994	LM	10
High-Performance Liquid Chromatographs (2):			
Waters 501 Pumps (2) with Waters 484 UV and 470 Fluorescence Detectors and LC 241 Autosampler	1991	LM	3
HP 1090M Series II with Microbore Terniary Pump system, HP 1046A Programmable Fluorescence Detector & Diode Array Detector .	1995	LM	3
Infrared Analyzer - Perkin-Elmer 1600 Series FTIR	1989	LM	4

MAJOR ANALYTICAL EQUIPMENT

GC/MS SEMIVOLATILE ORGANICS SAMPLE PREPARATION			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	7
Evaporators (4):			
Organomation N-Evap (1)	1989-1990	LM	7
Organomation S-Evap (2)	1990-1991	LM	7
Extractors (62):			
Continuous Liquid/Liquid Extractors (24)	1991	LM	7
Branson Model 450 Sonifier (2)	1991	LM	7
Tekmar Sonifier (2)	1994	LM	7
GPC-ABC Model Autoprep 1000	1995	LM	4
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	7

GC/MS SEMIVOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Chromatography Data Systems - HP Enviroquant (7)	1994	LM	5
Semivolatile GC/MS Systems (6):			
Hewlett-Packard 6890/5973 with HP 6890 Autosampler	1997	MM	3
Hewlett-Packard 5890/5970 with HP 7673 Autosampler (2)	1990,1994	MM	5
Hewlett-Packard 5890/5972 with HP 7673 Autosampler (3)	1993, 1994, 1998	MM	5

MAJOR ANALYTICAL EQUIPMENT

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (2)			
Mettler PE 160	1989	MM	9
Johnson Precisa 220M	1993	MM	9
Baxter Vortex Mixer	1989	LM	9
Extractors (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	3
TCLP Extractor - Tumbler (12 position)	1989	LM	3
HP Enviroquant Chromatography Data Systems (10)	1994	LM	9
Drying Ovens (2):			
Narco 420	1989	LM	9
VWR 1305 U	1991	LM	9
Sonic Water Bath - Branson Model 2200	1989	LM	9
Volatile GC/MS Systems (4):			
Hewlett-Packard 5890/5970	1989	MM	8
Tekmar 3000 Purge and Trap Concentrator	1995	LM	8
Dynatech ARCHON 5100 Autosampler	1996	LM	8
Hewlett-Packard 5890/5971	1991	MM	8
Tekmar-LSC-2000 Purge and Trap Concentrator	1992	LM	8
Dynatech ARCHON 5100 Autosampler	1995	LM	8
Hewlett-Packard 5890/5972A	1993	MM	8
Tekmar 3000 Purge and Trap Concentrator	1995	LM	8
Dynatech ARCHON 5100 Autosampler	1996	LM	8
Hewlett-Packard 6890/5973	1998	MM	4
Tekmar 3000 Purge and Trap Concentrator	1998	LM	4
Dynatech PTA-30 Autosampler	1998	LM	4
Volatile Gas Chromatographs (4):			
Varian 3300 GC with PID/ELCD detectors	1988	LM	6
O-I 4460A Purge and Trap Concentrator	1998	LM	6
Dynatech PTA-30 Autosampler	1988	LM	6
Hewlett-Packard 5890 Series II with PID/ELCD det.	1992	LM	6
Tekmar LSC-2000 Purge and Trap Concentrator	1988	LM	6
Dynatech PTA-30 Autosampler	1989	LM	6
Varian 3300 with PID/FID detectors	1989	LM	6
O-I 4460A Purge and Trap Concentrator	1989	LM	6
Dynatech PTA-30 Autosampler	1996	LM	6
Hewlett-Packard 5890 Series II with PID/FID det.	1991	LM	6
Tekmar LSC-2000 Purge and Trap Concentrator	1991	LM	6
Dynatech Archon 5100 Autosampler	1991	LM	6

MAJOR ANALYTICAL EQUIPMENT

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Purchased**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMs Sample Manager using ORACLE DBMS running on SEQUENT UNIX/DYNIX platform connected/linked on a frame relay WAN environment.	1994-1995	LM	3
2-LANs: HP Net Servers Pentium/Pentium II class. 1 for Administration and 1 for Data Acquisition. Network operating system: Novell® Advanced Netware Operating System. Total data acquisition capacity at 24GB with redundant tape and disk arrays. 3 NT Application servers, internal/external application.	1994-1998	LM	5
Appoximately 40-HP class Laserjet printers (various types from IIIs to SI IVs) linked via LAN.	1991-1998	LM	NA
Approximately 85 HP/Gateway PC/Workstations on LAN hooked up with 10BT/100BT and TCP/IP for LIMs Terminal Emulation.	1993-1998	LM	NA
Microsoft Office Professional as the base application for all PC/Workstations.	1994-1998	LM	NA
E-Mail with link to SMTP for internal/external messaging.	1994-1998	LM	NA
Standard EXCEL® reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1994-1996	LM	NA
Facsimile Machines 9600-14400 Baud, 11 Pages/Minute (4)	1991-1998	LM	NA
Dot Matrix NLQ (33)	1991-1992	LM	NA
Other assorted PCs (10)	1986-1994	LM	NA
Wyse 60 Terminals (20)	1991-1993	LM	NA
Thruput, MARRS/GARRS CLP software	1998	LM	NA
Telecation Enviroforms/Inorganic & Organic CLP software, SOW ILM04.0 & SOW OLM01.8 & 2.0	1995	LM	NA

NA: Not applicable. This equipment can be, and is, used by all staff.

APPENDIX C

DATA QUALITY CAPABILITIES

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accura[c] (%R
110.2	Color	Water	NA	NA	20	NA
120.1 / *SM* 2510B	Conductivity	Water	2 umhos/cm	0.3 umhos/cm	20	NA
130.2 / *SM* 2340C	Hardness as $CaCO_3$	Water	10 mg/L	4 mg/L	20	NA
150.1 / *SM* 4500-H⁺B	pH	Water	NA	NA	NA	NA
160.1 / *SM* 2540C	Solids, Total Dissolved (Filterable)	Water	5 mg/L	5 mg/L	20	NA
160.2 / *SM* 2540D	Solids, Total Suspended (Nonfilterable)	Water	5 mg/L	5 mg/L	20	NA
160.3 / *SM* 2540B	Solids, Total	Soil	NA (%)	NA (%)	20	NA
		Water	5 mg/L	5 mg/L	20	NA
160.4 / *SM* 2540E	Solids, Volatile	Soil	NA (%)	NA (%)	20	NA
		Water	5 mg/L	5 mg/L	20	NA
160.5 / *SM* 2540F	Solids, Settleable	Water	0.1 ml/L	NA	20	NA
180.1 / *SM* 2130B	Turbidity	Water	0.1 NTU	0.09 NTU	20	NA
300.0 / *SM* 4110B Ion Chromatography (After Extraction)	Bromide	Soil	2 mg/Kg		20	80-120[d]
	Chloride		2 mg/Kg		20	80-120[d]
	Fluoride		2 mg/Kg		20	80-120[d]
	Nitrate as Nitrogen		2 mg/Kg		20	80-120[d]
	Nitrite as Nitrogen		2 mg/Kg		20	80-120[d]
	Sulfate		2 mg/Kg		20	80-120[d]
300.0 / *SM* 4110B Ion Chromatography	Bromide	Water	0.2 mg/L	0.04 mg/L	20	80-1
	Chloride		0.2 mg/L	0.06 mg/L	20	80-120
	Fluoride		0.2 mg/L	0.05 mg/L	20	80-120[d]
	Nitrate as Nitrogen		0.2 mg/L	0.03 mg/L	20	80-120[d]
	Nitrite as Nitrogen		0.2 mg/L	0.04 mg/L	20	80-120[d]
	Sulfate		0.2 mg/L	0.06 mg/L	20	80-120[d]
305.1 / *SM* 2310B	Acidity as $CaCO_3$	Water	NA (mg/L)	NA (mg/L)	20	NA
310.1 / *SM* 2320B	Alkalinity as $CaCO_3$	Water	20 mg/L	0.7 mg/L	20	NA
325.3 / *SM* 4500-Cl⁻B	Chloride, Titrimetric	Water	0.2 mg/L	0.2 mg/L	20	75-125[b]
330.4 / *SM* 4500-Cl-F	Chlorine, Total Residual	Water	0.1 mg/L	0.07 mg/L	20	75-125[b]
335.1 / *SM* 4500-CN⁻G	Cyanides Amenable to Chlorination	Soil	0.2 mg/Kg		20	75-125[b]
		Water	0.01 mg/L	0.003 mg/L	20	75-125[b]
335.2 / *SM* 4500-CN⁻E	Cyanide, Total	Soil	0.2 mg/Kg		20	75-125[b]
		Water	0.01 mg/L	0.003 mg/L	20	75-125[b]
SM 4500-CN⁻I	Cyanide, Weak Acid Dissociable	Soil	0.2 mg/Kg		20	75-125[b]
		Water	0.01 mg/L	0.001 mg/L	20	75-125[b]
340.1 / *SM* 4500-F⁻B	Fluoride, Bellack Distillation	Soil	1 mg/Kg		20	75-125[b]
		Water	1 mg/L	0.6 mg/L	20	75-125[b]
340.2 / *SM* 4500-F⁻C	Fluoride	Soil	2 mg/Kg		20	75-125[b]
		Water	0.2 mg/L	0.02 mg/L	20	75-125[b]
350.3 / *SM* 4500-NH_3F	Ammonia as Nitrogen	Soil	0.2 mg/Kg		20	75-125[b]
		Water	0.05 mg/L	0.04 mg/L	20	75-
351.4	Nitrogen, Total Kjeldahl	Soil	10 mg/Kg		20	75-1
		Water	0.1 mg/L	0.1 mg/L	20	75-125[b]

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
353.2 / SM 4500-NO_3^--F	Nitrogen, Nitrate + Nitrite as Nitrogen	Soil Water	1 mg/Kg 0.2 mg/L	 0.01 mg/L	20 20	75-125[b] 75-125[b]
354.1 / *SM* 4500-NO_2^- - B	Nitrite as Nitrogen, Colorimetric	Water	0.01 mg/L	0.001 mg/L	20	75-125[b]
365.3 / *SM* 4500-PE	Orthophosphate as Phosphorus	Soil Water	(e) mg/Kg 0.01 mg/L	 0.002 mg/L	20 20	(e) 75 125[b]
365.3M 365.3 / *SM* 4500-PE	Phosphorus, Total	Soil Water	0.2 mg/Kg 0.01 mg/L	 0.009 mg/L	20 20	75-125[b] 75-125[b]
376.1 / *SM* 4500-S^{2-}D	Sulfide	Water	2 mg/L	0.3 mg/L	20	60-125[b]
377.1 / *SM* 4500-SO_3^{2-}B	Sulfite	Water	2 mg/L	0.5 mg/L	20	NA
SM 5550B	Tannin and Lignin	Water	0.2 mg/L	0.05 mg/L	20	75-125[b]
405.1 / *SM* 5210B	Biological Oxygen Demand	Water	4 mg/L	2 mg/L	20	NA
410.1 and 410.2 / *SM* 5220C	Chemical Oxygen Demand	Soil Water	50 mg/Kg 5 mg/L	 5 mg/L	20 20	75-125[b] 75-125[b]
ASTM D4129-82M 415.1	Total Organic Carbon	Soil Water	0.05% 0.5 mg/L	0.01% 0.07 mg/L	20 20	75-125[b] 85-115[b]
420.1 / *SM* 5530C	Phenolics, Total	Soil Water	0.5 mg/Kg 0.01 mg/L	 0.009 mg/L	20 20	75-125[b] 75-125[b]
425.1 / *SM* 5540C	Surfactants (MBAS)	Water	0.05 mg/L	0.05 mg/L	20	75-125[b]
ASTM D1498	Oxidation-Reduction Potential	Water	NA	NA	20	NA
1010	Flashpoint, Pensky-Marten	Soil Water	NA NA	NA NA	 20	 NA
1020	Flashpoint, Setaflash	Soil Water	NA NA	NA NA	 20	 NA
1110	Corrosivity	Liquid	NA	NA	20	NA
1650A	Absorbable Organic Halides	Water	10 ug/L	8 ug/L	20	71-116[d]
9010A	Cyanide, Total and Amenable	Soil Water	0.2 mg/Kg 0.01 mg/L	 0.003 mg/L	20 20	75-125[b] 75-125[b]
9020	Total Organic Halides	Water	10 ug/L	4 ug/L	20	75-125[b]
9030A 9030	Sulfides	Soil Water	4 mg/Kg 2 mg/L	 0.05 mg/L	20 20	75-125[b] 75-125[b]
9045C	pH	Soil Water	NA NA	NA NA	20 20	NA NA
9050	Specific Conductance	Water	2 mg/L	0.3 mg/L	20	NA
9060A	Total Organic Carbon	Water	0.5 mg/L	0.07 mg/L	20	75-125[b]
9066	Phenolics, Total	Soil Water	0.5 mg/Kg 0.01 mg/L	 0.004 mg/L	20 20	75-125[b] 75-125[b]
9095	Paint Filter Test	Soil	NA	NA	NA	NA
9252	Chloride, Titrimetric	Water	0.2 mg/L	0.2 mg/L	20	75-125[b]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%)
200.7 (ICP)	Aluminum	Water	50 ug/L	20 ug/L	20	75-1[illegible]
	Antimony		50 ug/L	30 ug/L	20	75-125[d]
	Barium		5 ug/L	3 ug/L	20	75-125[d]
	Beryllium		5 ug/L	1 ug/L	20	75-125[d]
	Boron		50 ug/L	30 ug/L	20	75-125[d]
	Cadmium		4 ug/L	3 ug/L	20	75-125[d]
	Calcium		50 ug/L	20 ug/L	20	75-125[d]
	Chromium		5 ug/L	4 ug/L	20	75-125[d]
	Cobalt		10 ug/L	9 ug/L	20	75-125[d]
	Copper		10 ug/L	6 ug/L	20	75-125[d]
	Iron		20 ug/L	20 ug/L	20	75-125[d]
	Magnesium		10 ug/L	8 ug/L	20	75-125[d]
	Manganese		5 ug/L	1 ug/L	20	75-125[d]
	Molybdenum		10 ug/L	6 ug/L	20	75-125[d]
	Nickel		20 ug/L	20 ug/L	20	75-125[d]
	Potassium		2000 ug/L	2000 ug/L	20	75-125[d]
	Silver		10 ug/L	5 ug/L	20	75-125[d]
	Sodium		100 ug/L	70 ug/L	20	75-125[d]
	Tin		50 ug/L	30 ug/L	20	75-[illegible]
	Vanadium		10 ug/L	3 ug/L	20	75-[illegible]
	Zinc		10 ug/L	4 ug/L	20	75-125[d]
206.2/200.9[f] (GFAA)	Arsenic		5 ug/L	1 ug/L	20	75-125[d]
239.2/200.9[f] (GFAA)	Lead		2 ug/L	2 ug/L	20	75-125[d]
245.1 (CVAA)	Mercury		0.5 ug/L	0.1 ug/L	20	85-115[e]
270.2/200.9[f] (GFAA)	Selenium		5 ug/L	1 ug/L	20	75-125[d]
279.2/200.9[f] (GFAA)	Thallium		5 ug/L	1 ug/L	20	75-125[d]
200.8 (ICP/MS)	Aluminum	Water	0.5 ug/L	0.3 ug/L	20	75-125[d]
	Antimony		0.02 ug/L	0.03 ug/L	20	75-125[d]
	Arsenic		0.5 ug/L	0.09 ug/L	20	75-125[d]
	Barium		0.03 ug/L	0.03 ug/L	20	75-125[d]
	Beryllium		0.02 ug/L	0.007 ug/L	20	75-125[d]
	Cadmium		0.02 ug/L	0.02ug/L	20	75-125[d]
	Chromium		0.2 ug/L	0.03 ug/L	20	75-125[d]
	Cobalt		0.02 ug/L	0.007 ug/L	20	75-125[d]
	Copper		0.1 ug/L	0.03 ug/L	20	75-125[d]
	Lead		0.02 ug/L	0.02 ug/L	20	75-125[d]
	Manganese		0.02 ug/L	0.02 ug/L	20	75-125[d]
	Molybdenum		0.03 ug/L	0.03 ug/L	20	75-125[d]
	Nickel		0.2 ug/L	0.05 ug/L	20	75-125[d]
	Selenium		1 ug/L	0.3 ug/L	20	7[illegible]
	Silver		0.02 ug/L	0.006 ug/L	20	75-[illegible]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Thallium		0.02 ug/L	0.007 ug/L	20	75-125[d]
	Vanadium		0.2 ug/L	0.02 ug/L	20	75-125[d]
	Zinc		0.5 ug/L	0.05 ug/L	20	75-125[d]
6010B (ICP)	Aluminum	Soil	10 mg/Kg	5 mg/Kg	30	75-125[d]
	Antimony		10 mg/Kg	5 mg/Kg	30	75-125[d]
	Barium		1 mg/Kg	0.6 mg/Kg	30	75-125[d]
	Beryllium		1 mg/Kg	0.2 mg/Kg	30	75-125[d]
	Boron		10 mg/Kg	5 mg/Kg	30	75-125[d]
	Cadmium		1 mg/Kg	0.6 mg/Kg	30	75-125[d]
	Calcium		10 mg/Kg	3 mg/Kg	30	75-125[d]
	Chromium		2 mg/Kg	0.7 mg/Kg	30	75-125[d]
	Cobalt		2 mg/Kg	2 mg/Kg	30	75-125[d]
	Copper		2 mg/Kg	2 mg/Kg	30	75-125[d]
	Iron		4 mg/Kg	4 mg/Kg	30	75-125[d]
	Lead		20 mg/Kg	5 mg/Kg	30	75-125[d]
	Magnesium		2 mg/Kg	2 mg/Kg	30	75-125[d]
	Manganese		1 mg/Kg	0.4 mg/Kg	30	75-125[d]
	Molybdenum		2 mg/Kg	1 mg/Kg	30	75-125[d]
	Nickel		10 mg/Kg	2 mg/Kg	30	75-125[d]
	Potassium		400 mg/Kg	400 mg/Kg	30	75-125[d]
	Silver		2 mg/Kg	0.8 mg/Kg	30	75-125[d]
	Sodium		20 mg/Kg	20 mg/Kg	30	75-125[d]
	Tin		10 mg/Kg	4 mg/Kg	30	75-125[d]
	Vanadium		2 mg/Kg	0.6 mg/Kg	30	75-125[d]
	Zinc		2 mg/Kg	0.8 mg/Kg	30	75-125[d]
7060A (GFAA)	Arsenic		1 mg/Kg	0.2 mg/Kg	30	60-130[b]
7195	Chromium, Hexavalent		0.5 mg/Kg			
7421 (GFAA)	Lead		1 mg/Kg	0.2 mg/Kg	30	60-130[b]
7471A (CVAA)	Mercury		0.2 mg/Kg	0.2 mg/Kg	30	60-130[b]
7740 (GFAA)	Selenium		1 mg/Kg	0.2 mg/Kg	30	60-130[b]
7841 (GFAA)	Thallium		1 mg/Kg	0.2 mg/Kg	30	60-130[b]
6010B (ICP)	Aluminum	Water	50 ug/L	20 ug/L	20	75-125[d]
	Antimony		50 ug/L	40 ug/L	20	75-125[d]
	Barium		5 ug/L	3 ug/L	20	75-125[d]
	Beryllium		5 ug/L	1 ug/L	20	75-125[d]
	Boron		50 ug/L	30 ug/L	20	75-125[d]
	Cadmium		4 ug/L	4 ug/L	20	75-125[d]
	Calcium		50 ug/L	8 ug/L	20	75-125[d]
	Chromium		5 ug/L	4 ug/L	20	75-125[d]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	Cobalt		10 ug/L	8 ug/L	20	75-1
	Copper		10 ug/L	5 ug/L	20	75-125[d]
	Iron		20 ug/L	20 ug/L	20	75-125[d]
	Magnesium		10 ug/L	8 ug/L	20	75-125[d]
	Manganese		5 ug/L	1 ug/L	20	75-125[d]
	Molybdenum		10 ug/L	5 ug/L	20	75-125[d]
	Nickel		20 ug/L	20 ug/L	20	75-125[d]
	Potassium		2000 ug/L	2000 ug/L	20	75-125[d]
	Silver		10 ug/L	4 ug/L	20	75-125[d]
	Sodium		100 ug/L	50 ug/L	20	75-125[d]
	Tin		50 ug/L	30 ug/L	20	75-125[d]
	Vanadium		10 ug/L	4 ug/L	20	75-125[d]
	Zinc		10 ug/L	4 ug/L	20	75-125[d]
7060A (GFAA)	Arsenic		5 ug/L	1 ug/L	20	75-125[d]
7421 (GFAA)	Lead		2 ug/L	2 ug/L	20	75-125[d]
7470A (CVAA)	Mercury		0.5 ug/L	0.1 ug/L	20	60-140[b]
7740 (GFAA)	Selenium		5 ug/L	1 ug/L	20	60-125[b]
7841 (GFAA)	Thallium		5 ug/L	1 ug/L	20	75-125[b]

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
413.1	Oil and Grease, Gravimetric	Water	5 mg/L	3 mg/L	30	
9071A	Oil and Grease, Gravimetric	Soil	100 mg/Kg	70 mg/Kg	40	
413.2	Oil and Grease, IR	Water	0.5 mg/L	0.2 mg/L	30	77-111
SM5520D/E	Oil and Grease, IR	Soil	25 mg/Kg	10 mg/Kg	40	72-111
418.1	Petroleum Hydrocarbons, Total Recoverable	Soil Water	25 mg/Kg 0.5 mg/L	10 mg/Kg 0.4 mg/L	40 30	72-115 77-111
504.1	1,2-Dibromoethane (EDB)	Drinking Water	0.01 ug/L	0.006 ug/L		60-140[d]
	1,2-Dibromo-3-chloropropane (DBCP)		0.02 ug/L	0.007 ug/L		60-140[d]
	Tetrachloro-*m* -xylene[g]		NA	NA	NA	20-165
515.1 Herbicides	Dalapon	Drinking Water	5 ug/L	0.2 ug/L	30	70-130[d]
	Dicamba		0.5 ug/L	0.05 ug/L	30	70-130[d]
	Dichloroprop		1 ug/L	0.05 ug/L	30	70-130[d]
	2,4-D		1 ug/L	0.09 ug/L	30	48-214
	Pentachlorophenol		0.5 ug/L	0.05 ug/L	30	70-130[d]
	2,4,5-TP (Silvex)		0.5 ug/L	0.06 ug/L	30	42-226
	2,4,5-T		0.5 ug/L	0.04 ug/L	30	68-166
	2,4-DB		5 ug/L	2 ug/L	30	70-130[d]
	Dinoseb		1 ug/L	0.05 ug/L	30	70-130[d]
	Picloram		1 ug/L	0.06 ug/L	30	70-130[d]
	2,4-Dichlorophenylacetic Acid[g]		NA	NA	NA	37-117
600/4-81-045 PCBs	Aroclor 1016	Oil	1 mg/Kg		30	
	Aroclor 1221		1 mg/Kg		30	
	Aroclor 1232		1 mg/Kg		30	
	Aroclor 1242		1 mg/Kg		30	31-173
	Aroclor 1248		1 mg/Kg		30	
	Aroclor 1254		1 mg/Kg		30	54-109
	Aroclor 1260		1 mg/Kg		30	49-120
	Decachlorobiphenyl[g]		NA	NA	NA	17-155
608 Chlorinated Pesticides and PCBs	alpha-BHC	Water	0.04 ug/L		30	
	gamma-BHC (Lindane)		0.04 ug/L		30	53-114
	beta-BHC		0.04 ug/L		30	
	Heptachlor		0.04 ug/L		30	20-116
	delta-BHC		0.04 ug/L		30	
	Aldrin		0.04 ug/L		30	20-117
	Heptachlor Epoxide		0.04 ug/L		30	
	Endosulfan I		0.04 ug/L		30	
	4,4'-DDE		0.04 ug/L		30	
	Dieldrin		0.04 ug/L		30	48-122
	Endrin		0.04 ug/L		30	56-120
	4,4'-DDD		0.04 ug/L		30	
	Endosulfan II		0.04 ug/L		30	
	4,4'-DDT		0.04 ug/L		30	53-121
	Endosulfan Sulfate		0.04 ug/L		30	
	Toxaphene		1 ug/L	0.2 ug/L	30	
	Chlordane		0.5 ug/L	0.09 ug/L	30	
	Aroclor 1016		0.2 ug/L		30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%RF
	Aroclor 1221		0.2 ug/L		30	
	Aroclor 1232		0.2 ug/L		30	
	Aroclor 1242		0.2 ug/L		30	
	Aroclor 1248		0.2 ug/L		30	
	Aroclor 1254		0.2 ug/L		30	
	Aroclor 1260		0.2 ug/L		30	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	20-100
	Decachlorobiphenyl[g]		NA	NA	NA	33-137
610 Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	5 ug/L		30	
	Acenaphthylene		5 ug/L		30	
	Acenaphthene		5 ug/L		30	24-122
	Fluorene		5 ug/L		30	
	Phenanthrene		5 ug/L		30	
	Anthracene		5 ug/L		30	
	Fluoranthene		5 ug/L		30	61-127
	Pyrene		5 ug/L		30	
	Benz(a)anthracene		5 ug/L		30	
	Chrysene		5 ug/L		30	
	Benzo(b+k)fluoranthene[h]		10 ug/L		30	
	Benzo(a)pyrene		5 ug/L		30	50-133
	Indeno(1,2,3-cd)pyrene and Dibenz(a,h)anthracene[h]		10 ug/L		30	
	Benzo(g,h,i)perylene		5 ug/L		30	
	p-Terphenyl[g]		NA	NA	NA	31-16
625 Base Neutral Acid Extractables	*N*-Nitrosodimethylamine	Water	10 ug/L	0.8 ug/L	30	
	Bis(2-chloroethyl) Ether		10 ug/L	0.7 ug/L	30	
	1,2-Dichlorobenzene		10 ug/L	0.7 ug/L	30	
	1,3-Dichlorobenzene		10 ug/L	0.6 ug/L	30	
	1,4-Dichlorobenzene		10 ug/L	0.6 ug/L	30	D-111
	Bis(2-chloroisopropyl) Ether		10 ug/L	0.7 ug/L	30	
	N-Nitrosodi-*n*-propylamine		10 ug/L	0.9 ug/L	30	43-100
	Hexachloroethane		10 ug/L	0.7 ug/L	30	
	Nitrobenzene		10 ug/L	0.7 ug/L	30	
	Isophorone		10 ug/L	0.9 ug/L	30	
	Bis(2-chloroethoxy)methane		10 ug/L	0.7 ug/L	30	
	1,2,4-Trichlorobenzene		10 ug/L	0.7 ug/L	30	46-90
	Naphthalene		10 ug/L	0.7 ug/L	30	
	Hexachlorobutadiene		10 ug/L	0.6 ug/L	30	
	2-Chloronaphthalene		10 ug/L	0.7 ug/L	30	
	Dimethyl Phthalate		10 ug/L	0.6 ug/L	30	
	Acenaphthylene		10 ug/L	0.7 ug/L	30	
	Acenaphthene		10 ug/L	0.7 ug/L	30	40-103
	2,4-Dinitrotoluene		10 ug/L	0.5 ug/L	30	49-98
	2,6-Dinitrotoluene		10 ug/L	0.8 ug/L	30	
	Diethyl Phthalate		10 ug/L	0.6 ug/L	30	
	4-Chlorophenyl Phenyl Ether		10 ug/L	0.7 ug/L	30	
	Fluorene		10 ug/L	0.7 ug/L	30	
	4-Bromophenyl Phenyl Ether		10 ug/L	0.7 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Hexachlorobenzene		10 ug/L	0.7 ug/L	30	
	Phenanthrene		10 ug/L	0.6 ug/L	30	
	Anthracene		10 ug/L	0.5 ug/L	30	
	Di-*n*-butyl Phthalate		10 ug/L	0.5 ug/L	30	
	Fluoranthene		10 ug/L	0.4 ug/L	30	
	Pyrene		10 ug/L	0.3 ug/L	30	45-120
	Butyl Benzyl Phthalate		10 ug/L	0.3 ug/L	30	
	3,3'-Dichlorobenzidine		25 ug/L	0.6 ug/L	30	
	Benz(a)anthracene		10 ug/L	0.5 ug/L	30	
	Bis(2-ethylhexyl) Phthalate		10 ug/L	0.7 ug/L	30	
	Chrysene		10 ug/L	0.4 ug/L	30	
	Di-*n*-octyl Phthalate		10 ug/L	2 ug/L	30	
	Benzo(b)fluoranthene		10 ug/L	0.4 ug/L	30	
	Benzo(k)fluoranthene		10 ug/L	0.7 ug/L	30	
	Benzo(a)pyrene		10 ug/L	0.5 ug/L	30	
	Indeno(1,2,3-c,d)pyrene		10 ug/L	0.6 ug/L	30	
	Dibenz(a,h)anthracene		10 ug/L	0.5 ug/L	30	
	Benzo(g,h,i)perylene		10 ug/L	0.6 ug/L	30	
	Phenol		10 ug/L	0.7 ug/L	30	4-106
	2-Chlorophenol		10 ug/L	0.8 ug/L	30	52-102
	2-Nitrophenol		10 ug/L	0.7 ug/L	30	
	2,4-Dimethylphenol		10 ug/L	2 ug/L	30	
	2,4-Dichlorophenol		10 ug/L	0.7 ug/L	30	
	4-Chloro-3-methylphenol		10 ug/L	0.7 ug/L	30	36-114
	2,4,6-Trichlorophenol		10 ug/L	0.6 ug/L	30	
	2,4-Dinitrophenol		25 ug/L	4 ug/L	30	
	4-Nitrophenol		25 ug/L	0.8 ug/L	30	8-121
	2-Methyl-4,6-dinitrophenol		25 ug/L	0.3 ug/L	30	
	Pentachlorophenol		25 ug/L	0.4 ug/L	30	D-126
	2-Fluorophenol[g]		NA	NA	NA	23-98
	Phenol-D6[g]		NA	NA	NA	D-114
	2,4,6-Tribromophenol[g]		NA	NA	NA	36-120
	Nitrobenzene-D5[g]		NA	NA	NA	42-100
	2-Fluorobiphenyl[g]		NA	NA	NA	45-99
	Terphenyl-D14[g]		NA	NA	NA	1-128
1653 Chlorinated Phenolics	4-Chlorophenol	Water	1.25 ug/L	0.04 ug/L	NA	NA
	2,4-Dichlorophenol		2.5 ug/L	0.06 ug/L	NA	NA
	2,6-Dichlorophenol		2.5 ug/L	0.06 ug/L	NA	NA
	2,4,5-Trichlorophenol		2.5 ug/L	0.04 ug/L	NA	NA
	2,4,6-Trichlorophenol		2.5 ug/L	0.07 ug/L	NA	NA
	2,3,4,6-Tetrachlorophenol		2.5 ug/L	0.04 ug/L	NA	NA
	Pentachlorophenol		5 ug/L	0.2 ug/L	NA	NA
	4-Chloroguaiacol		1.25 ug/L	0.04 ug/L	NA	NA
	3,4-Dichloroguaiacol		2.5 ug/L	0.05 ug/L	NA	NA
	4,5-Dichloroguaiacol		2.5 ug/L	0.07 ug/L	NA	NA
	4,6-Dichloroguaiacol		2.5 ug/L	0.05 ug/L	NA	NA
	3,4,5-Trichloroguaiacol		2.5 ug/L	0.06 ug/L	NA	NA

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	3,4,6-Trichloroguaiacol		2.5 ug/L	0.05 ug/L	NA	NA
	4,5,6-Trichloroguaiacol		2.5 ug/L	0.08 ug/L	NA	NA
	Tetrachloroguaiacol		5 ug/L	0.09 ug/L	NA	NA
	4-Chlorocatechol		1.25 ug/L	0.03 ug/L	NA	NA
	3,4-Dichlorocatechol		2.5 ug/L	0.05 ug/L	NA	NA
	3,6-Dichlorocatechol		2.5 ug/L	0.05 ug/L	NA	NA
	4,5-Dichlorocatechol		2.5 ug/L	0.04 ug/L	NA	NA
	3,4,5-Trichlorocatechol		5 ug/L	0.2 ug/L	NA	NA
	3,4,6-Trichlorocatechol		5 ug/L	0.2 ug/L	NA	NA
	Tetrachlorocatechol		5 ug/L	0.2 ug/L	NA	NA
	5-Chlorovanillin		2.5 ug/L	0.04 ug/L	NA	NA
	6-Chlorovanillin		2.5 ug/L	0.06 ug/L	NA	NA
	5,6-Dichlorovanillin		5 ug/L	0.1 ug/L	NA	NA
	2-Chlorosyringaldehyde		2.5 ug/L	0.08 ug/L	NA	NA
	2,6-Dichlorosyringaldehyde		5 ug/L	0.2 ug/L	NA	NA
	Trichlorosyringol		2.5 ug/L	0.09 ug/L	NA	NA
	2,4-Dichlorophenol-D3[g]		NA	NA	NA	27-143[d]
	4-Chloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	43-168[d]
	3,4,5-Trichlorophenol[g]		NA	NA	NA	24-167[d]
	5-Chlorovanillin-$^{13}C_6$[g]		NA	NA	NA	32-254[d]
	4,5-Dichlorocatechol-$^{13}C_6$[g]		NA	NA	NA	D-190[d]
	4,5,6-Trichloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	51-13[d]
	Pentachlorophenol-$^{13}C_6$[g]		NA	NA	NA	27-1
	Tetrachloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	27-16
	Tetrachlorocatechol-$^{13}C_6$[g]		NA	NA	NA	D-184[d]
8011	1,2-Dibromoethane (EDB)	Soil	1 ug/Kg		40	60-140[d]
	1,2-Dibromo-3-chloropropane(DBCP)		1 ug/Kg		40	60-140[d]
	Tetrachloro-*m* -xylene[g]		NA	NA	NA	24-137
8081A Chlorinated Pesticides	alpha-BHC	Soil	10 ug/Kg	0.3 ug/Kg	40	
	gamma-BHC (Lindane)		10 ug/Kg	0.2 ug/Kg	40	40-124
	beta-BHC		10 ug/Kg	0.2ug/Kg	40	
	Heptachlor		10 ug/Kg	0.4 ug/Kg	40	40-117
	delta-BHC		10 ug/Kg	0.3 ug/Kg	40	
	gamma-Chlordane		10 ug/Kg	0.2 ug/Kg	40	
	alpha-Chlordane		10 ug/Kg	0.2 ug/Kg	40	
	Aldrin		10 ug/Kg	0.4 ug/Kg	40	43-108
	Heptachlor Epoxide		10 ug/Kg	0.3 ug/Kg	40	
	Endosulfan I		10 ug/Kg	0.3 ug/Kg	40	
	4,4'-DDE		10 ug/Kg	0.2 ug/Kg	40	
	Dieldrin		10 ug/Kg	0.3 ug/Kg	40	42-127
	Endrin		10 ug/Kg	0.3 ug/Kg	40	46-123
	4,4'-DDD		10 ug/Kg	0.3 ug/Kg	40	
	Endosulfan II		10 ug/Kg	0.2 ug/Kg	40	
	4,4'-DDT		10 ug/Kg	0.3 ug/Kg	40	46-127
	Endrin Aldehyde		10 ug/Kg	0.3 ug/Kg	40	
	Endosulfan Sulfate		10 ug/Kg	0.2 ug/Kg	40	
	Methoxychlor		20 ug/Kg	0.1 ug/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Toxaphene		300 ug/Kg	50 ug/Kg	40	
	Tetrachloro-*m* -xylene[g]		NA	NA	NA	26-116
	Decachlorobiphenyl[g]		NA	NA	NA	33-143
8081A Chlorinated Pesticides	alpha-BHC	Water	0.04 ug/L	0.002 ug/L	30	
	gamma-BHC (Lindane)		0.04 ug/L	0.003 ug/L	30	53-114
	beta-BHC		0.04 ug/L	0.007 ug/L	30	
	Heptachlor		0.04 ug/L	0.003 ug/L	30	20-116
	delta-BHC		0.04 ug/L	0.004 ug/L	30	
	gamma-Chlordane		0.04 ug/L	0.002 ug/L	30	
	alpha-Chlordane		0.04 ug/L	0.005 ug/L	30	
	Aldrin		0.04 ug/L	0.005 ug/L	30	20-117
	Heptachlor Epoxide		0.04 ug/L	0.003 ug/L	30	
	Endosulfan I		0.04 ug/L	0.002 ug/L	30	
	4,4'-DDE		0.04 ug/L	0.007 ug/L	30	
	Dieldrin		0.04 ug/L	0.002 ug/L	30	48-122
	Endrin		0.04 ug/L	0.003 ug/L	30	56-120
	4,4'-DDD		0.04 ug/L	0.002 ug/L	30	
	Endosulfan II		0.04 ug/L	0.002 ug/L	30	
	4,4'-DDT		0.04 ug/L	0.003 ug/L	30	53-121
	Endrin Aldehyde		0.04 ug/L	0.002 ug/L	30	
	Endosulfan Sulfate		0.04 ug/L	0.004 ug/L	30	
	Methoxychlor		0.1 ug/L	0.002 ug/L	30	
	Toxaphene		1 ug/L	0.2 ug/L	30	
	Tetrachloro-*m* -xylene[g]		NA	NA	NA	20-100
	Decachlorobiphenyl[g]		NA	NA	NA	33-127
8080A, 8082 PCBs	Aroclor 1016	Soil	0.1 mg/Kg	0.02 mg/Kg	40	26-142
	Aroclor 1221		0.1 mg/Kg	0.03 mg/Kg	40	
	Aroclor 1232		0.1 mg/Kg	0.02 mg/Kg	40	
	Aroclor 1242		0.1 mg/Kg	0.03 mg/Kg	40	
	Aroclor 1248		0.1 mg/Kg	0.02 mg/Kg	40	
	Aroclor 1254		0.1 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1260		0.1 mg/Kg	0.02 mg/Kg	40	40-139
	Decachlorobiphenyl[g]		NA	NA	NA	42-130
8080A, 8082 PCBs	Aroclor 1016	Water	0.2 ug/L	0.04 ug/L	30	27-136
	Aroclor 1221		0.2 ug/L	0.09 ug/L	30	
	Aroclor 1232		0.2 ug/L	0.03 ug/L	30	
	Aroclor 1242		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1248		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1254		0.2 ug/L	0.03 ug/L	30	
	Aroclor 1260		0.2 ug/L	0.03 ug/L	30	27-136
	Decachlorobiphenyl[g]		NA	NA	NA	22-108
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Soil	1 ug/Kg	0.4 ug/Kg	40	
	2,2',5-Trichlorobiphenyl (PCB 18)		1 ug/Kg	0.2 ug/Kg	40	
	2,4,4'-Trichlorobiphenyl (PCB 28)		1 ug/Kg	0.09 ug/Kg	40	70-130[d]
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		1 ug/Kg	0.07 ug/Kg	40	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		1 ug/Kg	0.07 ug/Kg	40	70-130[d]
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		1 ug/Kg	0.09 ug/Kg	40	70-130[d]

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		1 ug/Kg	0.3 ug/Kg	40	70-130
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		1 ug/Kg	0.07 ug/Kg	40	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		1 ug/Kg	0.2 ug/Kg	40	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		1 ug/Kg	0.4 ug/Kg	40	70-130[d]
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		1 ug/Kg	0.1 ug/Kg	40	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		1 ug/Kg	0.07 ug/Kg	40	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		1 ug/Kg	0.07 ug/Kg	40	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		1 ug/Kg	0.2 ug/Kg	40	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		1 ug/Kg	0.2 ug/Kg	40	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		1 ug/Kg	0.3 ug/Kg	40	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		1 ug/Kg	0.2 ug/Kg	40	
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)		1 ug/Kg	0.09 ug/Kg	40	70-130[d]
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		1 ug/Kg	0.07 ug/Kg	40	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		1 ug/Kg	0.2 ug/Kg	40	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		1 ug/Kg	0.09 ug/Kg	40	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		1 ug/Kg	0.3 ug/Kg	40	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		1 ug/Kg	0.3 ug/Kg	40	70-130[d]
	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)		1 ug/Kg	0.08 ug/Kg	40	70-130[d]
	2,2',3,4,4',6,6'-Heptachlorobiphenyl (PCB 184)		1 ug/Kg	0.08 ug/Kg	40	70-130[d]
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB 187)		1 ug/Kg	0.2ug/Kg	40	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)		1 ug/Kg	0.08 ug/Kg	40	70-130
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB 195)		1 ug/Kg	0.07 ug/Kg	40	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB 206)		1 ug/Kg	0.07 ug/Kg	40	
	Decachlorobiphenyl (PCB 209)		1 ug/Kg	0.08 ug/Kg	40	70-130[d]
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	70-130[d]
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Water	5 ng/L	0.8 ng/L	30	
	2,2',5-Trichlorobiphenyl (PCB 18)		5 ng/L	0.7 ng/L	30	
	2,4,4'-Trichlorobiphenyl (PCB 28)		5 ng/L	0.6 ng/L	30	70-130[d]
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		5 ng/L	0.3 ng/L	30	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		5 ng/L	0.3 ng/L	30	70-130[d]
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		5 ng/L	0.7 ng/L	30	70-130[d]
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		5 ng/L	0.3 ng/L	30	70-130[d]
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		5 ng/L	0.2 ng/L	30	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		5 ng/L	0.8 ng/L	30	70-130[d]
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		5 ng/L	0.4 ng/L	30	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		5 ng/L	0.2 ng/L	30	70-130[d]
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		5 ng/L	0.3 ng/L	30	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		5 ng/L	0.2 ng/L	30	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		5 ng/L	0.2 ng/L	30	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		5 ng/L	0.2 ng/L	30	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		5 ng/L	0.3 ng/L	30	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		5 ng/L	0.2 ng/L	30	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		5 ng/L	0.2 ng/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		5 ng/L	0.2 ng/L	30	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		5 ng/L	0.4 ng/L	30	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		5 ng/L	0.3 ng/L	30	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		5 ng/L	0.3 ng/L	30	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		5 ng/L	0.2 ng/L	30	70-130[d]
	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,2',3,4,4',6,6'-Heptachlorobiphenyl (PCB 184)		5 ng/L	0.2 ng/L	30	70-130[d]
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB 187)		5 ng/L	0.2 ng/L	30	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)		5 ng/L	0.3 ng/L	30	70-130[d]
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB 195)		5 ng/L	0.3 ng/L	30	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB 206)		5 ng/L	0.5 ng/L	30	
	Decachlorobiphenyl (PCB 209)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	70-130[d]
8100 Polynuclear Aromatic Hydrocarbons	Naphthalene	Soil	0.5 mg/Kg		40	
	Acenaphthylene		0.5 mg/Kg		40	
	Acenaphthene		0.5 mg/Kg		40	49-111
	Fluorene		0.5 mg/Kg		40	
	Phenanthrene		0.5 mg/Kg		40	
	Anthracene		0.5 mg/Kg		40	
	Fluoranthene		0.5 mg/Kg		40	47-134
	Pyrene		0.5 mg/Kg		40	
	Benz(a)anthracene		0.5 mg/Kg		40	
	Chrysene		0.5 mg/Kg		40	
	Benzo(b+k)fluoranthene[h]		1.0 mg/Kg		40	
	Benzo(a)pyrene		0.5 mg/Kg		40	48-117
	Indeno(1,2,3-cd)pyrene and Dibenz(a.h)anthracene[h]		1.0 mg/Kg		40	
	Benzo(g,h,i)perylene		0.5 mg/Kg		40	
	p -Terphenyl[e]		NA	NA	NA	46-123
8100 Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	5 ug/L		30	
	Acenaphthylene		5 ug/L		30	
	Acenaphthene		5 ug/L		30	24-122
	Fluorene		5 ug/L		30	
	Phenanthrene		5 ug/L		30	
	Anthracene		5 ug/L		30	
	Fluoranthene		5 ug/L		30	61-127
	Pyrene		5 ug/L		30	
	Benz(a)anthracene		5 ug/L		30	
	Chrysene		5 ug/L		30	
	Benzo(b+k)fluoranthene[h]		10 ug/L		30	
	Benzo(a)pyrene		5 ug/L		30	50-133
	Indeno(1,2,3-cd)pyrene and Dibenz(a,h)anthracene[h]		10 ug/L		30	
	Benzo(g,h,i)perylene		5 ug/L		30	
	p -Terphenyl[g]		NA	NA	NA	31-162

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%RE
8141A Organo-phosphorus Pesticides	Azinphos Methyl	Soil	0.1 mg/Kg	0.002 mg/K	40	70-130
	Bolstar		0.1 mg/Kg	0.002 mg/K	40	70-130[d]
	Chlorpyrifos		0.1 mg/Kg	0.001 mg/K	40	70-130[d]
	Coumaphos		0.1 mg/Kg	0.002 mg/K	40	
	Demeton-O and Demeton-S[h]		0.1 mg/Kg	0.004 mg/K	40	
	Diazinon		0.1 mg/Kg	0.003 mg/K	40	70-130[d]
	Dichlorvos		0.1 mg/Kg	0.005 mg/K	40	
	Disulfoton		0.1 mg/Kg	0.002 mg/K	40	
	Ethoprop		0.1 mg/Kg	0.002 mg/K	40	
	Fensulfothion		0.1 mg/Kg	0.004 mg/K	40	
	Fenthion		0.1 mg/Kg	0.002 mg/K	40	
	Merphos		0.1 mg/Kg	0.003 mg/K	40	
	Mevinphos		0.1 mg/Kg	0.004 mg/K	40	
	Naled		0.1 mg/Kg	0.007 mg/K	40	
	Parathion Methyl		0.1 mg/Kg	0.001 mg/K	40	
	Phorate		0.1 mg/Kg	0.002 mg/K	40	
	Ronnel		0.1 mg/Kg	0.002 mg/K	40	70-130[d]
	Stirophos		0.1 mg/Kg	0.003 mg/K	40	
	Tokuthion (Prothiofos)		0.1 mg/Kg	0.002 mg/K	40	
	Trichloronate		0.1 mg/Kg	0.002 mg/K	40	70-130[d]
	Malathion		0.1 mg/Kg	0.001 mg/K	40	
	Triphenyl Phosphate[g]		NA	NA	NA	34-1
8141A Organo-phosphorus Pesticides	Azinphos Methyl	Water	1 ug/L	0.03 ug/L	30	70-130
	Bolstar		1 ug/L	0.03 ug/L	30	70-130[d]
	Chlorpyrifos		1 ug/L	0.03 ug/L	30	70-130[d]
	Coumaphos		1 ug/L	0.05 ug/L	30	
	Demeton-O and Demeton-S[h]		1 ug/L	0.05 ug/L	30	
	Diazinon		1 ug/L	0.04 ug/L	30	70-130[d]
	Dichlorvos		1 ug/L	0.03 ug/L	30	
	Disulfoton		1 ug/L	0.03 ug/L	30	
	Ethoprop		1 ug/L	0.03 ug/L	30	
	Fensulfothion		1 ug/L	0.05 ug/L	30	
	Fenthion		1 ug/L	0.05 ug/L	30	
	Merphos		1 ug/L	0.03 ug/L	30	
	Mevinphos		1 ug/L	0.04 ug/L	30	
	Naled		1 ug/L	0.03 ug/L	30	
	Parathion Methyl		1 ug/L	0.04 ug/L	30	
	Phorate		1 ug/L	0.03 ug/L	30	
	Ronnel		1 ug/L	0.04 ug/L	30	70-130[d]
	Stirophos		1 ug/L	0.03 ug/L	30	
	Tokuthion (Prothiofos)		1 ug/L	0.04 ug/L	30	
	Trichloronate		1 ug/L	0.03 ug/L	30	70-130[d]
	Malathion		1 ug/L	0.06 ug/L	30	
	Triphenyl Phosphate[g]		NA	NA	NA	18-1

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
8151A Chlorinated Herbicides	Dalapon	Soil	1 mg/Kg	0.2 mg/Kg	40	
	Dicamba		0.1 mg/Kg	0.007 mg/K	40	
	MCPA		20 mg/Kg	4 mg/Kg	40	
	MCPP		20 mg/Kg	4 mg/Kg	40	
	Dichloroprop		0.1 mg/Kg	0.04 mg/Kg	40	
	2,4-D		0.2 mg/Kg	0.03 mg/Kg	40	32-147
	2,4,5-TP (Silvex)		0.05 mg/Kg	0.005 mg/K	40	24-126
	2,4,5-T		0.05 mg/Kg	0.002 mg/K	40	23-137
	2,4-DB		0.5 mg/Kg	0.06 mg/Kg	40	
	Dinoseb		0.5 mg/Kg	0.03 mg/Kg	40	
	2,4-Dichlorophenylacetic Acid[g]		NA	NA	NA	22-122
8151A Chlorinated Herbicides	Dalapon	Water	5 ug/L	0.3 ug/L	30	
	Dicamba		0.5 ug/L	0.05 ug/L	30	
	MCPA		200 ug/L	5 ug/L	30	
	MCPP		200 ug/L	13 ug/L	30	
	Dichloroprop		0.6 ug/L	0.05 ug/L	30	
	2,4-D		1 ug/L	0.09 ug/L	30	44-145
	2,4,5-TP (Silvex)		0.2 ug/L	0.04 ug/L	30	36-146
	2,4,5-T		0.2 ug/L	0.04 ug/L	30	36-155
	2,4-DB		2 ug/L	2 ug/L	30	
	Dinoseb		2 ug/L	0.05 ug/L	30	
	Pentachlorophenol		2 ug/L	0.05 ug/L	30	
	4-Nitrophenol		2 ug/L	0.2 ug/L	30	
	2,4-Dichlorophenylacetic Acid[g]		NA	NA	NA	28-107
8151M Chlorinated Phenols	2,4,6-Trichlorophenol	Soil	5 ug/Kg	0.7 ug/Kg	40	
	Total Tetrachlorophenols		5 ug/Kg	2 ug/Kg	40	38-128
	Pentachlorophenol		5 ug/Kg	0.7 ug/Kg	40	32-126
	4-Bromo-2,6-dichlorophenol[g]		NA	NA	NA	19-129
8151M Chlorinated Phenols	2,4,6-Trichlorophenol	Water	0.5 ug/L	0.04 ug/L	30	
	Total Tetrachlorophenols		0.5 ug/L	0.09 ug/L	30	38-128
	Pentachlorophenol		0.5 ug/L	0.07 ug/L	30	43-124
	4-Bromo-2,6-dichlorophenol[g]		NA	NA	NA	42-122
8270C Base Neutral Acid Extractables	*N*-Nitrosodimethylamine	Soil	2 mg/Kg	0.06 mg/Kg	40	
	Aniline		1 mg/Kg	0.08 mg/Kg	40	
	Bis(2-chloroethyl) Ether		0.3 mg/Kg	0.08 mg/Kg	40	
	1,2-Dichlorobenzene		0.3 mg/Kg	0.07 mg/Kg	40	
	1,3-Dichlorobenzene		0.3 mg/Kg	0.07 mg/Kg	40	
	1,4-Dichlorobenzene		0.3 mg/Kg	0.07 mg/Kg	40	29-100
	Bis(2-chloroisopropyl) Ether		0.3 mg/Kg	0.06 mg/Kg	40	
	N-Nitrosodi-*n*-propylamine		0.3 mg/Kg	0.08 mg/Kg	40	26-112
	Hexachloroethane		0.3 mg/Kg	0.07 mg/Kg	40	
	Nitrobenzene		0.3 mg/Kg	0.08 mg/Kg	40	
	Isophorone		0.3 mg/Kg	0.09 mg/Kg	40	
	Bis(2-chloroethoxy)methane		0.3 mg/Kg	0.07 mg/Kg	40	
	1,2,4-Trichlorobenzene		0.3 mg/Kg	0.06 mg/Kg	40	31-109
	Naphthalene		0.3 mg/Kg	0.06 mg/Kg	40	
	4-Chloroaniline		0.3 mg/Kg	0.07 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R)
	Hexachlorobutadiene		0.3 mg/Kg	0.07 mg/Kg	40	
	2-Methylnaphthalene		0.3 mg/Kg	0.09 mg/Kg	40	
	Hexachlorocyclopentadiene		0.3 mg/Kg	0.07 mg/Kg	40	
	2-Chloronaphthalene		0.3 mg/Kg	0.05 mg/Kg	40	
	2-Nitroaniline		2 mg/Kg	0.05 mg/Kg	40	
	Dimethyl Phthalate		0.3 mg/Kg	0.1 mg/Kg	40	
	Acenaphthylene		0.3 mg/Kg	0.05 mg/Kg	40	
	3-Nitroaniline		2 mg/Kg	0.07 mg/Kg	40	
	Acenaphthene		0.3 mg/Kg	0.06 mg/Kg	40	46-105
	Dibenzofuran		0.3 mg/Kg	0.09 mg/Kg	40	
	2,4-Dinitrotoluene		0.3 mg/Kg	0.07 mg/Kg	40	54-114
	2,6-Dinitrotoluene		0.3 mg/Kg	0.05 mg/Kg	40	
	Diethyl Phthalate		0.3 mg/Kg	0.1 mg/Kg	40	
	4-Chlorophenyl Phenyl Ether		0.3 mg/Kg	0.09 mg/Kg	40	
	Fluorene		0.3 mg/Kg	0.07 mg/Kg	40	
	4-Nitroaniline		2 mg/Kg	0.05 mg/Kg	40	
	N-Nitrosodiphenylamine		0.3 mg/Kg	0.09 mg/Kg	40	
	4-Bromophenyl Phenyl Ether		0.3 mg/Kg	0.03 mg/Kg	40	
	Hexachlorobenzene		0.3 mg/Kg	0.06 mg/Kg	40	
	Phenanthrene		0.3 mg/Kg	0.08 mg/Kg	40	
	Anthracene		0.3 mg/Kg	0.05 mg/Kg	40	
	Di-*n* -butyl Phthalate		0.3 mg/Kg	0.07 mg/Kg	40	
	Fluoranthene		0.3 mg/Kg	0.08 mg/Kg	40	
	Pyrene		0.3 mg/Kg	0.06 mg/Kg	40	43-12
	Butyl Benzyl Phthalate		0.3 mg/Kg	0.07 mg/Kg	40	
	3,3'-Dichlorobenzidine		2 mg/Kg	0.04 mg/Kg	40	
	Benz(a)anthracene		0.3 mg/Kg	0.06 mg/Kg	40	
	Bis(2-ethylhexyl) Phthalate		0.3 mg/Kg	0.1 mg/Kg	40	
	Chrysene		0.3 mg/Kg	0.07 mg/Kg	40	
	Di-*n* -octyl Phthalate		0.3 mg/Kg	0.06 mg/Kg	40	
	Benzo(b)fluoranthene		0.3 mg/Kg	0.08 mg/Kg	40	
	Benzo(k)fluoranthene		0.3 mg/Kg	0.07 mg/Kg	40	
	Benzo(a)pyrene		0.3 mg/Kg	0.06 mg/Kg	40	
	Indeno(1,2,3-cd)pyrene		0.3 mg/Kg	0.04 mg/Kg	40	
	Dibenz(a,h)anthracene		0.3 mg/Kg	0.03 mg/Kg	40	
	Benzo(g,h,i)perylene		0.3 mg/Kg	0.04 mg/Kg	40	
	Phenol		0.3 mg/Kg	0.09 mg/Kg	40	32-97
	2-Chlorophenol		0.3 mg/Kg	0.06 mg/Kg	40	32-105
	Benzyl Alcohol		0.3 mg/Kg	0.1 mg/Kg	40	
	2-Methylphenol		0.3 mg/Kg	0.08 mg/Kg	40	
	3- and 4-Methylphenol[h]		0.3 mg/Kg	0.09 mg/Kg	40	
	2-Nitrophenol		0.3 mg/Kg	0.07 mg/Kg	40	
	2,4-Dimethylphenol		0.3 mg/Kg	0.05 mg/Kg	40	
	Benzoic Acid		2 mg/Kg	0.3 mg/Kg	40	
	2,4-Dichlorophenol		0.3 mg/Kg	0.08 mg/Kg	40	
	4-Chloro-3-methylphenol		0.3 mg/Kg	0.08 mg/Kg	40	31-
	2,4,6-Trichlorophenol		0.3 mg/Kg	0.09 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	2,4,5-Trichlorophenol		0.3 mg/Kg	0.08 mg/Kg	40	
	2,4-Dinitrophenol		2 mg/Kg	0.09 mg/Kg	40	
	4-Nitrophenol		2 mg/Kg	0.08 mg/Kg	40	21-133
	2-Methyl-4,6-dinitrophenol		2 mg/Kg	0.03 mg/Kg	40	
	Pentachlorophenol		2 mg/Kg	0.05 mg/Kg	40	38-107
	2-Fluorophenol[g]		NA	NA	NA	34-117
	Phenol-D6[g]		NA	NA	NA	30-104
	2,4,6-Tribromophenol[g]		NA	NA	NA	18-140
	Nitrobenzene-D5[g]		NA	NA	NA	21-115
	2-Fluorobiphenyl[g]		NA	NA	NA	34-117
	Terphenyl-D14[g]		NA	NA	NA	43-159
8270C Base Neutral Acid Extractables	*N*-Nitrosodimethylamine	Water	25 ug/L	0.8 ug/L	30	
	Pyridine		25 ug/L	2 ug/L	30	
	Aniline		25 ug/L	2 ug/L	30	
	Bis(2-chloroethyl) Ether		10 ug/L	0.7 ug/L	30	
	1,2-Dichlorobenzene		10 ug/L	0.7 ug/L	30	
	1,3-Dichlorobenzene		10 ug/L	0.6 ug/L	30	
	1,4-Dichlorobenzene		10 ug/L	0.6 ug/L	30	51-98
	Bis(2-chloroisopropyl) Ether		10 ug/L	0.7 ug/L	30	
	N-Nitrosodi-*n*-propylamine		10 ug/L	0.9 ug/L	30	43-114
	Hexachloroethane		10 ug/L	0.7 ug/L	30	
	Nitrobenzene		10 ug/L	0.7 ug/L	30	
	Isophorone		10 ug/L	0.9 ug/L	30	
	Bis(2-chloroethoxy)methane		10 ug/L	0.7 ug/L	30	
	1,2,4-Trichlorobenzene		10 ug/L	0.7 ug/L	30	42-113
	Naphthalene		10 ug/L	0.7 ug/L	30	
	4-Chloroaniline		10 ug/L	1 ug/L	30	
	Hexachlorobutadiene		10 ug/L	0.6 ug/L	30	
	2-Methylnaphthalene		10 ug/L	0.7 ug/L	30	
	Hexachlorocyclopentadiene		10 ug/L	4 ug/L	30	
	2-Chloronaphthalene		10 ug/L	0.7 ug/L	30	
	2-Nitroaniline		25 ug/L	0.6 ug/L	30	
	Dimethyl Phthalate		10 ug/L	0.6 ug/L	30	
	Acenaphthylene		10 ug/L	0.7 ug/L	30	
	3-Nitroaniline		25 ug/L	0.5 ug/L	30	
	Acenaphthene		10 ug/L	0.7 ug/L	30	50-114
	Dibenzofuran		10 ug/L	0.7 ug/L	30	
	2,4-Dinitrotoluene		10 ug/L	0.5 ug/L	30	55-123
	2,6-Dinitrotoluene		10 ug/L	0.8 ug/L	30	
	Diethyl Phthalate		10 ug/L	0.6 ug/L	30	
	4-Chlorophenyl Phenyl Ether		10 ug/L	0.7 ug/L	30	
	Fluorene		10 ug/L	0.7 ug/L	30	
	4-Nitroaniline		25 ug/L	0.6 ug/L	30	
	N-Nitrosodiphenylamine		10 ug/L	0.6 ug/L	30	
	4-Bromophenyl Phenyl Ether		10 ug/L	0.7 ug/L	30	
	Hexachlorobenzene		10 ug/L	0.7 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
	Anthracene		10 ug/L	0.5 ug/L	30	
	Carbazole		10 ug/L	0.6 ug/L	30	
	Di-*n* -butyl Phthalate		10 ug/L	0.5 ug/L	30	
	Fluoranthene		10 ug/L	0.4 ug/L	30	
	Pyrene		10 ug/L	0.3 ug/L	30	49-125
	Butyl Benzyl Phthalate		10 ug/L	0.3 ug/L	30	
	3,3'-Dichlorobenzidine		25 ug/L	0.6 ug/L	30	
	Benz(a)anthracene		10 ug/L	0.5 ug/L	30	
	Bis(2-ethylhexyl) Phthalate		10 ug/L	0.7 ug/L	30	
	Chrysene		10 ug/L	0.4 ug/L	30	
	Di-*n* -octyl Phthalate		10 ug/L	2 ug/L	30	
	Benzo(b)fluoranthene		10 ug/L	0.4 ug/L	30	
	Benzo(k)fluoranthene		10 ug/L	0.7 ug/L	30	
	Benzo(a)pyrene		10 ug/L	0.5 ug/L	30	
	Indeno(1,2,3-cd)pyrene		10 ug/L	0.6 ug/L	30	
	Dibenz(a,h)anthracene		10 ug/L	0.5 ug/L	30	
	Benzo(g,h,i)perylene		10 ug/L	0.6 ug/L	30	
	Phenol		10 ug/L	0.7 ug/L	30	37-102
	2-Chlorophenol		10 ug/L	0.8 ug/L	30	38-108
	Benzyl Alcohol		10 ug/L	0.7 ug/L	30	
	2-Methylphenol		10 ug/L	0.6 ug/L	30	
	3- and 4-Methylphenol[h]		10 ug/L	0.6 ug/L	30	
	2-Nitrophenol		10 ug/L	0.7 ug/L	30	
	2,4-Dimethylphenol		10 ug/L	2 ug/L	30	
	Benzoic Acid		25 ug/L	3 ug/L	30	
	2,4-Dichlorophenol		10 ug/L	0.7 ug/L	30	
	4-Chloro-3-methylphenol		10 ug/L	0.7 ug/L	30	39-120
	2,4,6-Trichlorophenol		10 ug/L	0.6 ug/L	30	
	2,4,5-Trichlorophenol		10 ug/L	0.6 ug/L	30	
	2,4-Dinitrophenol		25 ug/L	4 ug/L	30	
	4-Nitrophenol		25 ug/L	0.8 ug/L	30	15-147
	2-Methyl-4,6-dinitrophenol		25 ug/L	0.3 ug/L	30	
	Pentachlorophenol		25 ug/L	0.4 ug/L	30	34-126
	2-Fluorophenol[g]		NA	NA	NA	7-105
	Phenol-D6[g]		NA	NA	NA	22-118
	2,4,6-Tribromophenol[g]		NA	NA	NA	31-141
	Nitrobenzene-D5[g]		NA	NA	NA	32-123
	2-Fluorobiphenyl[g]		NA	NA	NA	42-122
	Terphenyl-D14[g]		NA	NA	NA	21-167
8310 Polynuclear Aromatic Hydrocarbons	Naphthalene	Soil	0.1 mg/Kg	0.04 mg/Kg	40	
	Acenaphthene		0.1 mg/Kg	0.02 mg/Kg	40	39-117
	Acenaphthylene		0.1 mg/Kg	0.1 mg/Kg	40	
	Fluorene		0.02 mg/Kg	0.01 mg/Kg	40	
	Phenanthrene		0.01 mg/Kg	0.004 mg/K	40	
	Anthracene		0.01 mg/Kg	0.004 mg/K	40	
	Fluoranthene		0.02 mg/Kg	0.008 mg/K	40	52-12
	Pyrene		0.02 mg/Kg	0.002 mg/K	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Benz(a)anthracene		0.01 mg/Kg	0.004 mg/K	40	
	Chrysene		0.01 mg/Kg	0.005 mg/K	40	
	Benzo(b)fluoranthene		0.02 mg/Kg	0.004 mg/K	40	
	Benzo(k)fluoranthene		0.01 mg/Kg	0.003 mg/K	40	
	Benzo(a)pyrene		0.01 mg/Kg	0.003 mg/K	40	44-123
	Dibenz(a,h)anthracene		0.01 mg/Kg	0.003 mg/K	40	
	Benzo(g,h,i)perylene		0.02 mg/Kg	0.005 mg/K	40	
	Indeno(1,2,3-cd)pyrene		0.01 mg/Kg	0.003 mg/K	40	
	p-Terphenyl[g]		NA	NA	NA	50-134
8310 Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	1 ug/L	0.4 ug/L	30	
	Acenaphthene		1 ug/L	0.4 ug/L	30	59-97
	Acenaphthylene		1 ug/L	0.3 ug/L	30	
	Fluorene		0.2 ug/L	0.05 ug/L	30	
	Phenanthrene		0.1 ug/L	0.01 ug/L	30	
	Anthracene		0.1 ug/L	0.02 ug/L	30	
	Fluoranthene		0.2 ug/L	0.07 ug/L	30	66-102
	Pyrene		0.2 ug/L	0.06 ug/L	30	
	Benz(a)anthracene		0.1 ug/L	0.03 ug/L	30	
	Chrysene		0.1 ug/L	0.02 ug/L	30	
	Benzo(b)fluoranthene		0.2 ug/L	0.05 ug/L	30	
	Benzo(k)fluoranthene		0.1 ug/L	0.04 ug/L	30	
	Benzo(a)pyrene		0.1 ug/L	0.03 ug/L	30	40-121
	Dibenz(a,h)anthracene		0.1 ug/L	0.1 ug/L	30	
	Benzo(g,h,i)perylene		0.2 ug/L	0.08 ug/L	30	
	Indeno(1,2,3-cd)pyrene		0.1 ug/L	0.05 ug/L	30	
	p-Terphenyl[g]		NA	NA	NA	32-142
8315	Formaldehyde	Soil	10 mg/Kg	0.6 mg/Kg	40	39-153[e]
8315	Formaldehyde	Water	50 ug/L	30 ug/L	30	39-153[e]
8330 Explosives	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	Soil	1 mg/Kg	0.05 mg/Kg	40	
	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)		1 mg/Kg	0.2 mg/Kg	40	
	1,3,5-Trinitrobenzene		1 mg/Kg	0.08 mg/Kg	40	
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)		1 mg/Kg	0.06 mg/Kg	40	
	1,3-Dinitrobenzene		1 mg/Kg	0.1 mg/Kg	40	
	2,4,6-Trinitrotoluene		1 mg/Kg	0.2 mg/Kg	40	66-127
	Nitrobenzene		1 mg/Kg	0.08 mg/Kg	40	71-101
	4-Amino-2,6-dinitrotoluene		1 mg/Kg	0.05 mg/Kg	40	
	2-Amino-4,6-dinitrotoluene		1 mg/Kg	0.1mg/Kg	40	
	2,6-Dinitrotoluene		1 mg/Kg	0.09 mg/Kg	40	
	2,4-Dinitrotoluene		1 mg/Kg	0.1 mg/Kg	40	77-106
	2-Nitrotoluene		1 mg/Kg	0.2 mg/Kg	40	
	4-Nitrotoluene		1 mg/Kg	0.2 mg/Kg	40	
	3-Nitrotoluene		1 mg/Kg	0.2 mg/Kg	40	
	4-Chloro-2-methylphenol[g]		NA	NA	NA	71-108
8330 Explosives	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	Water	1 ug/L	0.3 ug/L	30	
	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)		1 ug/L	0.2 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
	1,3,5-Trinitrobenzene		1 ug/L	0.2 ug/L	30	
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)		1 ug/L	0.6 ug/L	30	
	1,3-Dinitrobenzene		1 ug/L	0.3 ug/L	30	
	2,4,6-Trinitrotoluene		1 ug/L	0.5 ug/L	30	71-120
	Nitrobenzene		1 ug/L	0.4 ug/L	30	44-101
	4-Amino-2,6-dinitrotoluene		1 ug/L	0.5 ug/L	30	
	2-Amino-4,6-dinitrotoluene		1 ug/L	0.3 ug/L	30	
	2,6-Dinitrotoluene		1 ug/L	0.3 ug/L	30	
	2,4-Dinitrotoluene		1 ug/L	0.4 ug/L	30	76-112
	2-Nitrotoluene		1 ug/L	0.9 ug/L	30	
	4-Nitrotoluene		1 ug/L	1 ug/L	30	
	3-Nitrotoluene		1 ug/L	0.8 ug/L	30	
	4-Chloro-2-methylphenol[g]		NA	NA	NA	40-102
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Soil	5 ug/Kg	0.4 ug/Kg	40	
	1-Methylnaphthalene		5 ug/Kg	0.1 ug/Kg	40	
	2-Methylnaphthalene		5 ug/Kg	0.3 ug/Kg	40	
	Biphenyl		5 ug/Kg	0.2 ug/Kg	40	
	2,6-Dimethylnaphthalene		5 ug/Kg	0.2 ug/Kg	40	
	Acenaphthylene		5 ug/Kg	0.2 ug/kg	40	
	Dibenzofuran		5 ug/Kg	0.3 ug/Kg	40	
	Acenaphthene		5 ug/Kg	0.2 ug/Kg	40	36-119
	2,3,5-Trimethylnaphthalene		5 ug/Kg	0.2 ug/Kg	40	
	Fluorene		5 ug/Kg	0.3 ug/Kg	40	
	Phenanthrene		5 ug/Kg	0.4 ug/Kg	40	
	Anthracene		5 ug/Kg	0.2 ug/Kg	40	
	1-Methylphenanthrene		5 ug/Kg	0.3 ug/Kg	40	
	Fluoranthene		5 ug/Kg	0.6 ug/Kg	40	
	Pyrene		5 ug/Kg	0.5 ug/Kg	40	37-137
	Benz(a)anthracene		5 ug/Kg	0.4 ug/Kg	40	
	Chrysene		5 ug/Kg	0.5 ug/Kg	40	
	Benzo(b)fluoranthene		5 ug/Kg	0.8 ug/kg	40	
	Benzo(k)fluoranthene		5 ug/Kg	0.4 ug/Kg	40	
	Benzo(a)pyrene		5 ug/Kg	0.3 ug/Kg	40	24-137
	Benzo(e)pyrene		5 ug/Kg	0.3 ug/Kg	40	
	Indeno(1,2,3-cd)pyrene		5 ug/Kg	0.6 ug/Kg	40	
	Dibenz(a,h)anthracene		5 ug/Kg	0.4 ug/Kg	40	
	Benzo(g,h,i)perylene		5 ug/Kg	0.6 ug/Kg	40	
	Fluorene-d10[g]		NA	NA	NA	13-144
	Fluoranthene-d10[g]		NA	NA	NA	13-144
	Terphenyl-d14[g]		NA	NA	NA	15-145
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	0.02 ug/L	0.005 ug/L	30	
	1-Methylnaphthalene		0.02 ug/L	0.004 ug/L	30	
	2-Methylnaphthalene		0.02 ug/L	0.004 ug/L	30	
	Biphenyl		0.02 ug/L	0.004 ug/L	30	
	2,6-Dimethylnaphthalene		0.02 ug/L	0.004 ug/L	30	
	Acenaphthylene		0.02 ug/L	0.004 ug/L	30	
	Dibenzofuran		0.02 ug/L	0.007 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Acenaphthene		0.02 ug/L	0.004 ug/L	30	40-102
	2,3,5-Trimethylnaphthalene		0.02 ug/L	0.005 ug/L	30	
	Fluorene		0.02 ug/L	0.006 ug/L	30	
	Phenanthrene		0.02 ug/L	0.02 ug/L	30	
	Anthracene		0.02 ug/L	0.02 ug/L	30	
	1-Methylphenanthrene		0.02 ug/L	0.003 ug/L	30	
	Fluoranthene		0.02 ug/L	0.009 ug/L	30	
	Pyrene		0.02 ug/L	0.007 ug/L	30	39-128
	Benz(a)anthracene		0.02 ug/L	0.01 ug/L	30	
	Chrysene		0.02 ug/L	0.01 ug/L	30	
	Benzo(b)fluoranthene		0.02 ug/L	0.01 ug/L	30	
	Benzo(k)fluoranthene		0.02 ug/L	0.02 ug/L	30	
	Benzo(a)pyrene		0.02 ug/L	0.02 ug/L	30	47-114
	Benzo(e)pyrene		0.02 ug/L	0.003 ug/L	30	
	Indeno(1,2,3-cd)pyrene		0.02 ug/L	0.02 ug/L	30	
	Dibenz(a,h)anthracene		0.02 ug/L	0.02 ug/L	30	
	Benzo(g,h,i)perylene		0.02 ug/L	0.02 ug/L	30	
	Fluorene-d10[g]		NA	NA	NA	39-124
	Fluoranthene-d10[g]		NA	NA	NA	39-124
	Terphenyl-d14[g]		NA	NA	NA	44-127
GC/FPD Organotin Compounds	Tributyltin	Soil	1 ug/Kg	0.4 ug/Kg	40	27-162
	Dibutyltin		1 ug/Kg	0.4 ug/Kg	40	8-161
	Monobutyltin		1 ug/Kg	0.3 ug/Kg	40	8-161
	Tetrabutyltin		1 ug/Kg	0.5 ug/Kg	40	27-162
	Tripropyltin[g]		NA	NA	NA	18-125
	Tripentyltin[g]		NA	NA	NA	28-122
GC/FPD Organotin Compounds	Tributyltin	Water	0.05 ug/L	0.005 ug/L	30	23-131
	Dibutyltin		0.05 ug/L	0.005 ug/L	30	16-118
	Monobutyltin		0.05 ug/L	0.01 ug/L	30	17-128
	Tripropyltin[g]		NA	NA	NA	21-107
	Tripentyltin[g]		NA	NA	NA	21-116
AK 102	Diesel Range Petroleum Hydrocarbons	Soil	10 mg/Kg	2 mg/Kg	40	60-120[d]
CA-TPH-D			10 mg/Kg	2 mg/Kg	40	60-120
OR-TPH-D			10 mg/Kg	2 mg/Kg	40	60-120
WA-TPH-D			25 mg/Kg	2 mg/Kg	40	60-120
	o-Terphenyl[g]		NA	NA	NA	56-116
AK 102	Diesel Range Petroleum Hydrocarbons	Water	100 ug/L	30 ug/L	30	46-108
CA-TPH-D			50 ug/L	20 ug/L	30	46-108
OR-TPH-D			50 ug/L	20 ug/L	30	46-108
WA-TPH-D			250 ug/L	20 ug/L	30	46-108
	o-Terphenyl[g]		NA	NA	NA	59-110
AK 103	Residual Range Petroleum Hydrocarbons	Soil	10 mg/Kg		40	60-100[d]
	n-Triacontane[g]		NA	NA	NA	50-150[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%P
524.2	Dichlorodifluoromethane (CFC 12)	Drinking Water	0.5 ug/L	0.2 ug/L	NA	80-
	Chloromethane		0.5 ug/L	0.2 ug/L	NA	80-12
	Vinyl Chloride		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Bromomethane		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	Chloroethane		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,1-Dichloroethene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Methylene Chloride		0.5 ug/L	0.06 ug/L	NA	80-120[d]
	trans-1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	2,2-Dichloropropane		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	cis-1,2-Dichloroethene		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,1-Dichloroethane		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	Chloroform		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	Bromochloromethane		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,1-Dichloropropene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Carbon Tetrachloride		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Benzene		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,2-Dichloroethane		0.5 ug/L	0.08 ug/L	NA	80-
	Trichloroethene (TCE)		0.5 ug/L	0.2 ug/L	NA	80-
	1,2-Dichloropropane		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	Bromodichloromethane		0.5 ug/L	0.06 ug/L	NA	80-120[d]
	Dibromomethane		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	cis-1,3-Dichloropropene		0.5 ug/L	0.07 ug/L	NA	80-120[d]
	Toluene		0.5 ug/L	0.08 ug/L	NA	80-120[d]
	trans-1,3-Dichloropropene		0.5 ug/L	0.06 ug/L	NA	80-120[d]
	1,1,2-Trichloroethane		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Tetrachloroethene (PCE)		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,3-Dichloropropane		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	1,2,3-Trichlorobenzene		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	Dibromochloromethane		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,2-Dibromoethane (EDB)		0.5 ug/L	0.07 ug/L	NA	80-120[d]
	Chlorobenzene		0.5 ug/L	0.08 ug/L	NA	80-120[d]
	Ethylbenzene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,1,1,2-Tetrachloroethane		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	Styrene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Total Xylenes		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Bromoform		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Isopropylbenzene		0.5 ug/L	0.2 ug/L	NA	80
	1,1,2,2-Tetrachloroethane		0.5 ug/L	0.08 ug/L	NA	80-
	1,2,3-Trichloropropane		0.5 ug/L	0.07 ug/L	NA	80-120[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Bromobenzene		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	n -Propylbenzene		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	1,3,5-Trimethylbenzene		0.5 ug/L	0.08 ug/L	NA	80-120[d]
	2-Chlorotoluene		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	4-Chlorotoluene		0.5 ug/L	0.06 ug/L	NA	80-120[d]
	tert -Butylbenzene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,2,4-Trimethylbenzene		0.5 ug/L	0.07 ug/L	NA	80-120[d]
	sec -Butylbenzene		0.5 ug/L	0.1 ug/L	NA	80-120[d]
	4-Isopropyltoluene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,3-Dichlorobenzene		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,4-Dichlorobenzene		0.5 ug/L	0.07 ug/L	NA	80-120[d]
	n -Butylbenzene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,2-Dichlorobenzene		0.5 ug/L	0.08 ug/L	NA	80-120[d]
	1,2-Dibromo-3-chloropropane (DBCP)		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	1,2,4-Trichlorobenzene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Hexachlorobutadiene		0.5 ug/L	0.2 ug/L	NA	80-120[d]
	Naphthalene		0.5 ug/L	0.09 ug/L	NA	80-120[d]
	1,2-Dichlorobenzene-D4[g]		0.5 ug/L	NA	NA	80-120[d]
	4-Bromofluorobenzene[g]		0.5 ug/L	NA	NA	80-120[d]
601	Bromodichloromethane	Water	0.5 ug/L	0.4 ug/L	30	42-172[d]
	Bromoform		0.5 ug/L	0.3 ug/L	30	13-159[d]
	Bromomethane		0.5 ug/L	0.3 ug/L	30	D-144[d]
	Carbon Tetrachloride		0.5 ug/L	0.4 ug/L	30	43-143[d]
	Chlorobenzene		0.5 ug/L	0.4 ug/L	30	38-150[d]
	Chloroethane		0.5 ug/L	0.3 ug/L	30	46-137[d]
	2-Chloroethyl Vinyl Ether		5 ug/L	0.3 ug/L	30	14-186[d]
	Chloroform		0.5 ug/L	0.4 ug/L	30	49-133[d]
	Chloromethane		1 ug/L	0.4 ug/L	30	D-193[d]
	Dibromochloromethane		0.5 ug/L	0.3 ug/L	30	24-191[d]
	1,2-Dichlorobenzene		1 ug/L	0.3 ug/L	30	D-208[d]
	1,3-Dichlorobenzene		1 ug/L	0.2 ug/L	30	7-187[d]
	1,4-Dichlorobenzene		1 ug/L	0.2 ug/L	30	42-143[d]
	Dichlorodifluoromethane (CFC 12)		1 ug/L	0.2 ug/L	30	
	1,1-Dichloroethane		0.5 ug/L	0.4 ug/L	30	47-132[d]
	1,2-Dichloroethane		0.5 ug/L	0.4 ug/L	30	51-147[d]
	1,1-Dichloroethene		0.5 ug/L	0.4 ug/L	30	32-165[d]
	trans -1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	30	38-155[d]
	1,2-Dichloropropane		0.5 ug/L	0.4 ug/L	30	44-156[d]
	cis-1,3-Dichloropropene		0.5 ug/L	0.3 ug/L	30	22-178[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	trans-1,3-Dichloropropene		0.5 ug/L	0.4 ug/L	30	22-1
	Methylene Chloride		5 ug/L	0.5 ug/L	30	25-162[d]
	1,1,2,2-Tetrachloroethane		0.5 ug/L	0.4 ug/L	30	8-184[d]
	Tetrachloroethene (PCE)		0.5 ug/L	0.3 ug/L	30	57-157[d]
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.4 ug/L	30	41-138[d]
	1,1,2-Trichloroethane		0.5 ug/L	0.4 ug/L	30	39-136[d]
	Trichloroethene (TCE)		0.5 ug/L	0.4 ug/L	30	71-139[d]
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.3 ug/L	30	21-156[d]
	Vinyl Chloride		0.5 ug/L	0.3 ug/L	30	28-163[d]
	Bromochloromethane[g]		NA	NA	NA	38-131
602	Benzene	Water	0.5 ug/L	0.3 ug/L	30	67-131[d]
	Chlorobenzene		0.5 ug/L	0.4 ug/L	30	55-135[d]
	1,2-Dichlorobenzene		1 ug/L	0.3 ug/L	30	37-154[d]
	1,3-Dichlorobenzene		1 ug/L	0.2 ug/L	30	50-141[d]
	1,4-Dichlorobenzene		1 ug/L	0.2 ug/L	30	42-143[d]
	Toluene		1 ug/L	0.5 ug/L	30	61-129[d]
	Ethylbenzene		1 ug/L	0.4 ug/L	30	64-126[d]
	Total Xylenes		1 ug/L	0.5 ug/L	30	
	1,4-Difluorobenzene[g]		NA	NA	NA	70
624	Chloromethane	Water	10 ug/L	0.9 ug/L	30	D-2
	Vinyl Chloride		10 ug/L	0.9 ug/L	30	D-251[d]
	Bromomethane		10 ug/L	0.7 ug/L	30	D-242[d]
	Chloroethane		10 ug/L	0.7 ug/L	30	14-230[d]
	Trichlorofluoromethane (CFC 11)		10 ug/L	0.9 ug/L	30	17-181[d]
	Trichlorotrifluoroethane (CFC 113)		10 ug/L	0.9 ug/L	30	
	1,1-Dichloroethene		5 ug/L	0.8 ug/L	30	70-130[b]
	Carbon Disulfide		100 ug/L	0.6 ug/L	30	
	Methylene Chloride		5 ug/L	0.4 ug/L	30	D-221[d]
	trans-1,2-Dichloroethene		5 ug/L	0.7 ug/L	30	54-156[d]
	cis-1,2-Dichloroethene		5 ug/L	0.5 ug/L	30	
	2-Butanone (MEK)		100 ug/L	5 ug/L	30	
	1,1-Dichloroethane		5 ug/L	0.7 ug/L	30	59-155[d]
	Chloroform		5 ug/L	0.6 ug/L	30	51-138[d]
	1,1,1-Trichloroethane (TCA)		5 ug/L	0.9 ug/L	30	52-162[d]
	Carbon Tetrachloride		5 ug/L	0.8 ug/L	30	70-140[d]
	Benzene		5 ug/L	0.7 ug/L	30	70-130[b]
	1,2-Dichloroethane		5 ug/L	0.4 ug/L	30	49-155[d]
	Vinyl Acetate		50 ug/L	3 ug/L	30	
	Trichloroethene (TCE)		5 ug/L	0.5 ug/L	30	70-
	1,2-Dichloropropane		5 ug/L	0.7 ug/L	30	D-210[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Bromodichloromethane		5 ug/L	0.5 ug/L	30	35-155[d]
	2-Chloroethyl Vinyl Ether		10 ug/L	0.4 ug/L	30	D-305[d]
	trans -1,3-Dichloropropene		5 ug/L	0.4 ug/L	30	17-183[d]
	2-Hexanone		50 ug/L	4 ug/L	30	
	4-Methyl-2-pentanone (MIBK)		50 ug/L	3 ug/L	30	
	Toluene		5 ug/L	0.8 ug/L	30	70-130[b]
	cis -1,3-Dichloropropene		5 ug/L	0.4 ug/L	30	D-227[d]
	1,1,2-Trichloroethane		5 ug/L	0.5 ug/L	30	52-150[d]
	Tetrachloroethene (PCE)		5 ug/L	0.9 ug/L	30	64-148[d]
	Dibromochloromethane		5 ug/L	2 ug/L	30	53-149[d]
	Chlorobenzene		5 ug/L	0.7 ug/L	30	70-130[b]
	Ethylbenzene		5 ug/L	0.7 ug/L	30	37-162[d]
	Styrene		5 ug/L	0.5 ug/L	30	
	Total Xylenes		5 ug/L	2 ug/L	30	
	Bromoform		5 ug/L	0.3 ug/L	30	45-169[d]
	1,1,2,2-Tetrachloroethane		5 ug/L	0.3 ug/L	30	46-157[d]
	1,3-Dichlorobenzene		5 ug/L	0.6 ug/L	30	59-156[d]
	1,4-Dichlorobenzene		5 ug/L	0.6 ug/L	30	18-190[d]
	1,2-Dichlorobenzene		5 ug/L	0.6 ug/L	30	18-190[d]
	1,2-Dichloroethane-D4[g]		NA	NA	NA	87-117
	Toluene-D8[g]		NA	NA	NA	93-109
	4-Bromofluorobenzene[g]		NA	NA	NA	85-111
1624	Acetone	Water	50 ug/L	2 ug/L	30	55-145[d]
	Methylene Chloride		10 ug/L	0.5 ug/L	30	D-250[d]
	2-Butanone (MEK)		50 ug/L	0.5 ug/L	30	42-158[d]
	Chloroform		10 ug/L	0.5 ug/L	30	40-150[d]
	Acetone-D6[g]		NA	NA	NA	35-165[d]
	Methylene Chloride-D2[g]		NA	NA	NA	D-316[d]
	2-Butanone-D5[g]		NA	NA	NA	36-164[d]
	Chloroform-^{13}C[g]		NA	NA	NA	18-172[d]
8021B Volatile Aromatics (Low Level)	Benzene	Soil	5 ug/Kg	0.7 ug/Kg	40	61-119
	Toluene		5 ug/Kg	0.7 ug/Kg	40	59-118
	Ethylbenzene		5 ug/Kg	0.7 ug/Kg	40	51-132
	Total Xylenes		5 ug/Kg	2 ug/Kg	40	
	1,4-Difluorobenzene[g]		NA	NA	NA	52-123
8021B Volatile Halogenated and Aromatic Organics	Dichlorodifluoromethane (CFC 12)	Soil	0.1 mg/Kg		40	
	Chloromethane		0.1 mg/Kg		40	
	Vinyl Chloride		0.05 mg/Kg		40	
	Bromomethane		0.05 mg/Kg		40	
	Chloroethane		0.05 mg/Kg		40	
	Trichlorofluoromethane (CFC 11)		0.05 mg/Kg		40	
	1,1-Dichloroethene		0.05 mg/Kg		40	32-165
	Trichlorotrifluoroethane (CFC 113)		0.05 mg/Kg		40	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	Methylene Chloride		0.5 mg/Kg		40	
	trans-1,2-Dichloroethene		0.05 mg/Kg		40	
	cis -1,2-Dichloroethene		0.05 mg/Kg		40	
	1,1-Dichloroethane		0.05 mg/Kg		40	
	Chloroform		0.05 mg/Kg		40	
	1,1,1-Trichloroethane (TCA)		0.05 mg/Kg		40	
	Carbon Tetrachloride		0.05 mg/Kg		40	
	1,2-Dichloroethane		0.05 mg/Kg		40	
	Trichloroethene (TCE)		0.05 mg/Kg		40	71-139
	1,2-Dichloropropane		0.05 mg/Kg		40	
	Bromodichloromethane		0.05 mg/Kg		40	
	2-Chloroethyl Vinyl Ether		0.5 mg/Kg		40	
	trans -1,3-Dichloropropene		0.05 mg/Kg		40	
	cis -1,3-Dichloropropene		0.05 mg/Kg		40	
	1,1,2-Trichloroethane		0.05 mg/Kg		40	
	Tetrachloroethene (PCE)		0.05 mg/Kg		40	57-157
	Dibromochloromethane		0.05 mg/Kg		40	
	Chlorobenzene		0.05 mg/Kg	0.01 mg/Kg	40	
	Bromoform		0.05 mg/Kg		40	
	1,1,2,2-Tetrachloroethane		0.05 mg/Kg		40	
	1,3-Dichlorobenzene		0.1 mg/Kg	0.02 mg/Kg	40	
	1,4-Dichlorobenzene		0.1 mg/Kg	0.01 mg/Kg	40	
	1,2-Dichlorobenzene		0.1 mg/Kg	0.02 mg/Kg	40	
	Benzene		0.05 mg/Kg	0.01 mg/Kg	40	74-130
	Toluene		0.1 mg/Kg	0.01 mg/Kg	40	74-132
	Ethylbenzene		0.1 mg/Kg	0.01 mg/Kg	40	32-160
	Total Xylenes		0.1 mg/Kg	0.02 mg/Kg	40	
	Methyl *tert* -Butyl Ether (MTBE)		0.5 mg/Kg		40	
	1,4-Difluorobenzene[g]		NA	NA	NA	70-130[d]
	Bromochloromethane[g]		NA	NA	NA	54-123
8021B Volatile Halogenated and Aromatic Organics	Dichlorodifluoromethane (CFC 12)	Water	1 ug/L	0.2 ug/L	30	
	Chloromethane		1 ug/L	0.4 ug/L	30	
	Vinyl Chloride		0.5 ug/L	0.3 ug/L	30	
	Bromomethane		0.5 ug/L	0.3 ug/L	30	
	Chloroethane		0.5 ug/L	0.3 ug/L	30	
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.3 ug/L	30	
	1,1-Dichloroethene		0.5 ug/L	0.4 ug/L	30	32-165
	Trichlorotrifluoroethane (CFC 113)		0.5 ug/L	0.3 ug/L	30	
	Methylene Chloride		5 ug/L	0.5 ug/L	30	
	trans -1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	30	
	cis -1,2-Dichloroethene		0.5 ug/L	0.3 ug/L	30	
	1,1-Dichloroethane		0.5 ug/L	0.4 ug/L	30	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Chloroform		0.5 ug/L	0.4 ug/L	30	
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.4 ug/L	30	
	Carbon Tetrachloride		0.5 ug/L	0.4 ug/L	30	
	1,2-Dichloroethane		0.5 ug/L	0.4 ug/L	30	
	Trichloroethene (TCE)		0.5 ug/L	0.4 ug/L	30	71-139
	1,2-Dichloropropane		0.5 ug/L	0.4 ug/L	30	
	Bromodichloromethane		0.5 ug/L	0.4 ug/L	30	
	2-Chloroethyl Vinyl Ether		5 ug/L	0.3 ug/L	30	
	trans -1,3-Dichloropropene		0.5 ug/L	0.4 ug/L	30	
	cis -1,3-Dichloropropene		0.5 ug/L	0.3 ug/L	30	
	1,1,2-Trichloroethane		0.5 ug/L	0.4 ug/L	30	
	Tetrachloroethene (PCE)		0.5 ug/L	0.3 ug/L	30	57-157
	Dibromochloromethane		0.5 ug/L	0.3 ug/L	30	
	Chlorobenzene		0.5 ug/L	0.2 ug/L	30	
	Bromoform		0.5 ug/L	0.3 ug/L	30	
	1,1,2,2-Tetrachloroethane		0.5 ug/L	0.4 ug/L	30	
	1,3-Dichlorobenzene		1 ug/L	0.1 ug/L	30	
	1,4-Dichlorobenzene		1 ug/L	0.2 ug/L	30	
	1,2-Dichlorobenzene		1 ug/L	0.3 ug/L	30	
	Benzene		0.5 ug/L	0.2 ug/L	30	74-130
	Toluene		1 ug/L	0.1 ug/L	30	74-132
	Ethylbenzene		1 ug/L	0.2 ug/L	30	32-160
	Total Xylenes		1 ug/L	0.2 ug/L	30	
	Methyl *tert* -Butyl Ether (MTBE)		5 ug/L		30	
	1,4-Difluorobenzene[g]		NA	NA	NA	70-130[d]
	Bromochloromethane[g]		NA	NA	NA	73-146
8260B Volatile Organics	Dichlorodifluoromethane (CFC 12)	Soil	5 ug/Kg	2 ug/Kg	40	
	Chloromethane		5 ug/Kg	2 ug/Kg	40	
	Vinyl Chloride		5 ug/Kg	2 ug/Kg	40	
	Bromomethane		5 ug/Kg	2 ug/Kg	40	
	Chloroethane		5 ug/Kg	2 ug/Kg	40	
	Trichlorofluoromethane (CFC 11)		5 ug/Kg	3 ug/Kg	40	
	Trichlorotrifluoromethane (CFC 113)		5 ug/Kg	2 ug/Kg	40	
	Acetone		50 ug/Kg	1 ug/Kg	40	
	1,1-Dichloroethene		5 ug/Kg	2 ug/Kg	40	73-118
	Carbon Disulfide		5 ug/Kg	2 ug/Kg	40	
	Methylene Chloride		10 ug/Kg	2 ug/Kg	40	
	trans -1,2-Dichloroethene		5 ug/Kg	2 ug/Kg	40	
	1,1-Dichloroethane		5 ug/Kg	2 ug/Kg	40	
	Methyl tert-butyl ether		5 ug/Kg	1 ug/Kg	40	
	2-Butanone (MEK)		20 ug/Kg	2 ug/Kg	40	
	2,2-Dichloropropane		5 ug/Kg	2 ug/Kg	40	
	cis -1,2-Dichloroethene		5 ug/Kg	2 ug/Kg	40	
	Chloroform		5 ug/Kg	2 ug/Kg	40	
	Bromochloromethane		5 ug/Kg	0.7 ug/Kg	40	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	1,1,1-Trichloroethane (TCA)		5 ug/Kg	2 ug/Kg	40	
	1,1-Dichloropropene		5 ug/Kg	2 ug/Kg	40	
	Carbon Tetrachloride		5 ug/Kg	2 ug/Kg	40	
	1,2-Dichloroethane		5 ug/Kg	0.8 ug/Kg	40	
	Benzene		5 ug/Kg	2 ug/Kg	40	78-116
	Trichloroethene (TCE)		5 ug/Kg	2 ug/Kg	40	79-119
	1,2-Dichloropropane		5 ug/Kg	2 ug/Kg	40	
	Bromodichloromethane		5 ug/Kg	0.9 ug/Kg	40	
	Dibromomethane		5 ug/Kg	0.8 ug/Kg	40	
	2-Hexanone		20 ug/Kg	2 ug/Kg	40	
	cis -1,3-Dichloropropene		5 ug/Kg	0.7 ug/Kg	40	
	Toluene		5 ug/Kg	2 ug/Kg	40	77-118
	trans -1,3-Dichloropropene		5 ug/Kg	0.7 ug/Kg	40	
	1,1,2-Trichloroethane		5 ug/Kg	0.7 ug/Kg	40	
	4-Methyl-2-pentanone (MIBK)		20 ug/Kg	2 ug/Kg	40	
	1,3-Dichloropropane		5 ug/Kg	0.6 ug/Kg	40	
	Tetrachloroethene (PCE)		5 ug/Kg	2 ug/Kg	40	
	Dibromochloromethane		5 ug/Kg	0.7 ug/Kg	40	
	1,2-Dibromoethane (EDB)		20 ug/Kg	0.6 ug/Kg	40	
	Chlorobenzene		5 ug/Kg	1 ug/Kg	40	80-117
	1,1,1,2-Tetrachloroethane		5 ug/Kg	1 ug/Kg	40	
	Ethylbenzene		5 ug/Kg	2 ug/Kg	40	
	Total Xylenes		5 ug/Kg	4 ug/Kg	40	
	Styrene		5 ug/Kg	0.8 ug/Kg	40	
	Bromoform		5 ug/Kg	0.6 ug/Kg	40	
	Isopropylbenzene		20 ug/Kg	2 ug/Kg	40	
	1,1,2,2-Tetrachloroethane		5 ug/Kg	0.9 ug/Kg	40	
	1,2,3-Trichloropropane		5 ug/Kg	1 ug/Kg	40	
	Bromobenzene		5 ug/Kg	0.8 ug/Kg	40	
	n -Propylbenzene		20 ug/Kg	2 ug/Kg	40	
	2-Chlorotoluene		20 ug/Kg	2 ug/Kg	40	
	4-Chlorotoluene		20 ug/Kg	2 ug/Kg	40	
	1,3,5-Trimethylbenzene		20 ug/Kg	2 ug/Kg	40	
	tert -Butylbenzene		20 ug/Kg	2 ug/Kg	40	
	1,2,4-Trimethylbenzene		20 ug/Kg	2 ug/Kg	40	
	sec -Butylbenzene		20 ug/Kg	2 ug/Kg	40	
	1,3-Dichlorobenzene		5 ug/Kg	0.9 ug/Kg	40	
	4-Isopropyltoluene		20 ug/Kg	2 ug/Kg	40	
	1,4-Dichlorobenzene		5 ug/Kg	0.8 ug/Kg	40	
	n -Butylbenzene		20 ug/Kg	2 ug/Kg	40	
	1,2-Dichlorobenzene		5 ug/Kg	0.6 ug/Kg	40	79-120
	1,2-Dibromo-3-chloropropane (DBCP)		20 ug/Kg	2 ug/Kg	40	
	1,2,4-Trichlorobenzene		20 ug/Kg	0.7 ug/Kg	40	
	1,2,3-Trichlorobenzene		20 ug/Kg	0.6 ug/Kg	40	
	Naphthalene		20 ug/Kg	0.7 ug/Kg	40	57-[illegible]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
	Hexachlorobutadiene		20 ug/Kg	2 ug/Kg	40	
	Toluene-D8[g]		NA	NA	NA	85-109
	4-Bromofluorobenzene[g]		NA	NA	NA	49-131
	Dibromofluoromethane[g]		NA	NA	NA	75-132
8260B Volatile Organics	Dichlorodifluoromethane (CFC 12)	Water	0.5 ug/L	0.2 ug/L	30	
	Chloromethane		0.5 ug/L	0.2 ug/L	30	
	Vinyl Chloride		0.5 ug/L	0.3 ug/L	30	
	Bromomethane		0.5 ug/L	0.3 ug/L	30	
	Chloroethane		0.5 ug/L	0.2 ug/L	30	
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.2 ug/L	30	
	Trichlorotrifluoromethane (CFC 113)		0.5 ug/L	0.2 ug/L	30	
	Acetone		20 ug/L	8 ug/L	30	
	1,1-Dichloroethene		0.5 ug/L	0.2 ug/L	30	62-148
	Methyl tert-butyl ether		0.5 ug/L	0.2 ug/L	30	
	Carbon Disulfide		0.5 ug/L	0.2 ug/L	30	
	Methylene Chloride		1 ug/L	0.3 ug/L	30	
	trans -1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	30	
	1,1-Dichloroethane		0.5 ug/L	0.2 ug/L	30	
	2-Butanone (MEK)		20 ug/L	6 ug/L	30	
	2,2-Dichloropropane		0.5 ug/L	0.3 ug/L	30	
	cis -1,2-Dichloroethene		0.5 ug/L	0.3 ug/L	30	
	Chloroform		0.5 ug/L	0.2 ug/L	30	
	Bromochloromethane		0.5 ug/L	0.2 ug/L	30	
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.3 ug/L	30	
	1,1-Dichloropropene		0.5 ug/L	0.2 ug/L	30	
	Carbon Tetrachloride		0.5 ug/L	0.3 ug/L	30	
	1,2-Dichloroethane		0.5 ug/L	0.2 ug/L	30	
	Benzene		0.5 ug/L	0.2 ug/L	30	77-114
	Trichloroethene (TCE)		0.5 ug/L	0.3 ug/L	30	69-124
	1,2-Dichloropropane		0.5 ug/L	0.2 ug/L	30	
	Bromodichloromethane		0.5 ug/L	0.2 ug/L	30	
	Dibromomethane		0.5 ug/L	0.1 ug/L	30	
	2-Hexanone		20 ug/L	6 ug/L	30	
	cis -1,3-Dichloropropene		0.5 ug/L	0.2 ug/L	30	
	Toluene		0.5 ug/L	0.2 ug/L	30	75-118
	trans -1,3-Dichloropropene		0.5 ug/L	0.2 ug/L	30	
	1,1,2-Trichloroethane		0.5 ug/L	0.3 ug/L	30	
	4-Methyl-2-pentanone (MIBK)		20 ug/L	7 ug/L	30	
	1,3-Dichloropropane		0.5 ug/L	0.2 ug/L	30	
	Tetrachloroethene (PCE)		0.5 ug/L	0.2 ug/L	30	
	Dibromochloromethane		0.5 ug/L	0.2 ug/L	30	
	1,2-Dibromoethane (EDB)		2 ug/L	0.2 ug/L	30	
	Chlorobenzene		0.5 ug/L	0.2 ug/L	30	79-110
	1,1,1,2-Tetrachloroethane		0.5 ug/L	0.2 ug/L	30	
	Ethylbenzene		0.5 ug/L	0.2 ug/L	30	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%R
	Total Xylenes		0.5 ug/L	0.4 ug/L	30	
	Styrene		0.5 ug/L	0.2 ug/L	30	
	Bromoform		0.5 ug/L	0.2 ug/L	30	
	Isopropylbenzene		2 ug/L	0.2 ug/L	30	
	1,1,2,2-Tetrachloroethane		0.5 ug/L	0.2 ug/L	30	
	1,2,3-Trichloropropane		0.5 ug/L	0.3 ug/L	30	
	Bromobenzene		0.5 ug/L	0.2 ug/L	30	
	n -Propylbenzene		2 ug/L	0.2 ug/L	30	
	2-Chlorotoluene		2 ug/L	0.2 ug/L	30	
	4-Chlorotoluene		2 ug/L	0.2 ug/L	30	
	1,3,5-Trimethylbenzene		2 ug/L	0.2 ug/L	30	
	tert -Butylbenzene		2 ug/L	0.2 ug/L	30	
	1,2,4-Trimethylbenzene		2 ug/L	0.2 ug/L	30	
	sec -Butylbenzene		2 ug/L	0.2 ug/L	30	
	1,3-Dichlorobenzene		0.5 ug/L	0.2 ug/L	30	
	4-Isopropyltoluene		2 ug/L	0.2 ug/L	30	
	1,4-Dichlorobenzene		0.5 ug/L	0.2 ug/L	30	
	n -Butylbenzene		2 ug/L	0.2 ug/L	30	
	1,2-Dichlorobenzene		0.5 ug/L	0.2 ug/L	30	80-108
	1,2-Dibromo-3-chloropropane (DBCP)		2 ug/L	0.5 ug/L	30	
	1,2,4-Trichlorobenzene		2 ug/L	0.2 ug/L	30	
	1,2,3-Trichlorobenzene		2 ug/L	0.2 ug/L	30	
	Naphthalene		2 ug/L	0.2 ug/L	30	64-
	Hexachlorobutadiene		2 ug/L	0.2 ug/L	30	
	Toluene-D8[g]		NA	NA	NA	89-111
	4-Bromofluorobenzene[g]		NA	NA	NA	79-126
	Dibromofluoromethane[g]		NA	NA	NA	89-111
AK 101	Gasoline Range Petroleum Hydrocarbons	Soil	5 mg/Kg	1 mg/Kg	40	82-155
CA-TPH-G			5 mg/Kg	2 mg/Kg	40	82-155
OR-TPH-G			10 mg/Kg	4 mg/Kg	40	82-155
WA-TPH-G			5 mg/Kg	2 mg/Kg	40	82-155
	1,4-Difluorobenzene[g]		NA	NA	NA	52-123
AK 101	Gasoline Range Petroleum Hydrocarbons	Water	50 ug/L	40 ug/L	30	60-120[d]
CA-TPH-G			50 ug/L	40 ug/L	30	76-138
OR-TPH-G			50 ug/L	30 ug/L	30	76-138
WA-TPH-G			50 ug/L	40 ug/L	30	76-138
	1,4-Difluorobenzene[g]		NA	NA	NA	70-130[d]

MICROBIOLOGICAL ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
SM 9215B	Heterotrophic Plate Count (Pour)	Water	1 CFU/mL	NA	NA	NA
SM 9215C	Heterotrophic Plate Count (Spread)		1 CFU/mL	NA	NA	NA
SM 9221B	Coliform, Total (MPN)		2 MPN/100mL[i]	NA	NA	NA
SM 9221E	Coliform, Fecal (MPN)		2 MPN/100mL[i]	NA	NA	NA
SM 9223B	Coliform (Colilert)		Presence/100mL	NA	NA	NA
SM 9230B	Enterococcus		2 MPN/100mL	NA	NA	NA
SM 9230B	Fecal Streptococcus		2 MPN/100mL	NA	NA	NA
SM 10200H	Chlorophyll A		0.8 mg/L	0.3 mg/L	20	NA

Legend: NA = Not Applicable PCBs = Polychlorinated Biphenyls

a See Section 18 for list of references. Unless noted, the listed methods are EPA methods: *SM* = *Standard Methods for the Examination of Water and Wastewater*; ASTM = American Society for Testing and Materials. Microbiology methods are *Standard Methods for the Examination of Water and Wastewater*, 18th edition.

b In-house limits, unless footnoted.

c Laboratory control sample (LCS) limits. Statistically derived values unless footnoted.

d Method specified control limits.

e Method MRLs and matrix spike percent recovery data vary according to the method of extraction.

f Either method may be used, depending on project requirements.

g Surrogate compound

h These compounds coelute; therefore, results are reported as the combined concentration.

i MRL is 1.1 MPN/100mL for drinking water samples (10 tube method).

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Before every use
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride SIE	Store in storage solution	Between uses
Ammonia SIE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed

Instrument	Activity	Frequency
Flow Injection Analyzer	Check valve flares	Monthly
	Check valve ports	Monthly
	Check pump tubing	Monthly
	Check light counts	Monthly
	Check flow cell flares	Quarterly
	Change bulb	Every six months
	Check manifold tubing	Every six months
	Check T's and connectors	Every six months
Ion Chromatographs	Change column bed supports	Monthly or as needed
	Clean column	Monthly or as needed
	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	Weekly
	Clean RF contact strip	Weekly
	Inspect nebulizer, spray chamber, and torch	Weekly, clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Infrared Spectrophotometer, Fourier Transform	Clean sample cells	Daily, or as needed
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure to high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	Every six months
	Change pump diaphragm	Every six months
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean NPD	Quarterly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	Every six months
	Oil wick	Every six months
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Replenish ELCD solvents	Weekly
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	Every six months
	Oil wick	Every six months

STANDARD OPERATING PROCEDURE

BUTYLTINS
SOC-BUTYL
Revision 1
February 28, 1997

Approved By:

Supervisor	3-2-97 Date
QA Coordinator	2-28-97 Date
Laboratory Manager	3-5-97 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Standard Operating Procedure

for

BUTYLTINS

1. SCOPE AND APPLICATION

This method is used to determine the concentrations of butyltins in water, sediment and tissue. Tributyltin chloride (Bu_3SnCl) as well as butyltin trichloride ($BuSnCl_3$) dibutyltin dichloride (Bu_2SnCl_2), and tetrabutyltin (Bu_4Sn) can be determined by this method. The butyltin chloride salts of tributyltin, dibutyltin and monobutyltin are reported as butyltin cations. The current reporting limit for each of these analytes is listed in Table 1.

2. METHOD SUMMARY

The Butyltins method is a modification of procedures outlined in various papers (Unger, et. al; Krone, et.al.) in performing the extraction, derivitization, and analysis of mono-, di-, tri-, and tetrabutyltin chloride. This method involves a 0.1% tropolone in methylene chloride extraction of the analytes of intent from an acidified sample followed by a Grignard reaction of the hexane extract with Hexylmagnesium bromide (HxMgBr). The extract is then eluted through silica and alumina cartridge columns for cleanup and then analyzed by GC/FPD with a 610nm bandwidth filter. The extraction procedure used is outlined in the SOP "SOC-OSWT".

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Sulfur compounds cause the most direct interference with this determination. Copper shavings or powder is used to help remove the sulfur that might otherwise interfere with the flame photometric detector.

5. SAFETY

The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.2 Water and soil samples must be iced or refrigerated at 4 °C from time of collection until extraction.

6.3 Water samples should be extracted within 7 days and soil samples should be extracted within 14 days. Studies have not been done to determine sample or extract stability.

7. STANDARDS AND REAGENTS

7.1 Neat standards may be purchased from Aldrich or Alfa.

$BuSnCl_3$	Aldrich	20,105-7
Bu_2SnCl_2	Aldrich	20,549-4
Bu_3SnCl	Aldrich	T5,020-2
Bu_4Sn	Aldrich	T600-8
Pe_3SnCl	Aldrich	37,135-1
Pr_3SnCl	Alfa	71122

7.2 Stock Solutions - 5000 µg/mL

7.2.1 Butyltin Stock

A stock solution of Tetrabutyltin, Tributyltin, Dibutyltin, and Monobutyltin cations is prepared by weighing out the following amounts:

Tetrabutyltin	50 mg
Tributyltin chloride	56.1 mg
Dibutyltin dichloride	65.2 mg

Butyltin trichloride 80.2 mg

Dilute to 10 mL in DCM. Replace this solution yearly, or sooner if there are signs of degradation.

7.2.2 Surrogate Stock

Stock solutions of each surrogate (tripropyltin chloride and tripentyltin chloride) are prepared individually by weighing 50 mg and diluting to 10 mL in DCM. These solutions are good for 1 year.

7.3 Working Standards - 100 µg/mL

7.3.1 Weekly Butyltin Standard - Appropriately dilute the 5000 µg/mL stock solution for butyltins in Acetone (5000 µg/mL x 200 µL/10 mL of acetone). Replace weekly.

7.3.2 Weekly Surrogate Standard - Appropriately dilute each 5000 µg/mL stock solutions of tripentyltin chloride and tripropyltin chloride into one mixture in acetone (5000 µg/mL x 200 µL/10 mL of acetone). Replace weekly.

7.4 Daily Standards - 5 µg/mL

7.4.1 Daily Butyltin Spike Standard - Appropriately dilute weekly butyltin standard in acetone (100 µg/mL x 0.5 mL/10 mL). Prepare on each day of extraction.

7.4.2 Daily Butyltin Surrogate Spike - Appropriately dilute weekly butyltin standard in acetone (100 µg/mL x 0.5 mL/10 mL). Prepare on each day of extraction.

7.5 Calibration Stock Standard

The calibration stock standard is prepared by diluting 1 mL each of the working surrogate solution and the working spike solution to 20 µg/mL in 5 mL Hexane (100 µg/mL x 1 mL/5 mL) and derivitizing with Griguard reagent. This standard is usually prepared at the same time that a sample set is derivatized.

7.6 Calibration Standards

Calibration standards are prepared by diluting the calibration stock solution into appropriate levels. A minimum of three levels (five is recommended) is used to demonstrate linearity by a least square fit curve or by the average response factor. Recommended concentrations are: 0, 5, 10, 50, 100, 500, 1000, and 2000 µg/L as

cations. Each standard will include the surrogates. Currently, no independent calibration verification standard source is available. To convert the butyltin cation concentration to the corresponding chloride salt concentration, divide the cation conc. by the following correction factors:

Tributyltin	=	0.8910
Dibutyltin	=	0.7665
Monobutyltin	=	0.6230

7.7 Store all standards (neat and in solution) in a freezer (<4°C).

8. EQUIPMENT AND OPERATING CONDITIONS

8.1 Gas Chromatograph (GC) equipped with a chromatography data system and a flame photometric detector (FPD), HP 5890 with Enviroquant.

8.2 A 610nm bandwidth filter for the FPD (purchased from Corion, Holliston, MA, 508-429-5065, Part No. 510-610-F)

8.3 Recommended GC Parameters

8.3.1 Column: Restek RTX-5 megabore 30m x 0.53mm I.D., 1.0 µm film thickness (Cat. No. 10255) or equivalent.

8.3.2 Recommended Operating Conditions

Temp 1	150°C	Carrier flow (column)	8ml/min.
Time 1	1 minute	Makeup flow (detector)	22ml/min.
Rate	12°C/minute	Hydrogen flow (detector)	175ml/min.
Temp 2	300°C	Air flow (detector)	100ml/min.
Time 2	4.5 minutes		

9. PROCEDURE

9.1 Calibration

Calibrate the system immediately prior to conducting any analyses. Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area) versus the concentration in the standard. The ratio of the response to the amount injected, defined as the calibration factor (CF), may be calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average calibration factor may be used in place of a calibration curve.

If %RSD exceeds 20%, the analyst may elect to plot a linear or quadratic regression curve. Print plot and evaluate curve by quantitating hits near the MRL to check for gross error. Curves should be forced through zero.

The following should be used as warning limits:

- linear regression R^2 value < 0.990
- % RSD on the average RF's exceed 50%
- curve cannot be forced through zero without introducing significant error (>50% of true value at low point of curve)

If any of the above warning limits are exceeded, the analyst should do the following:

- Assess instrument's ability to attain MRL for this analyte. There needs to be sufficient sensitivity.
- Recalculate low, high and CCV points using new curve. The value calculated should be within 50% of true value on the low point, 15% on the CCV and 30% on the high point. If this is not the case, the curve needs to be rerun for that analyte.
- Obtain a secondary opinion from a senior analyst or supervisor.

9.2 Calibration Verification

9.2.1 The start of any sequence must include a CCV checked against the Initial Calibration (curve or average calibration factors). For any analyte to "pass" it must be within ± 25% D of the expected response. For this specific "passing" analyte, this CCV is valid for 24 hours and is considered an

"Opening CCV". An Opening CCV must be run prior to (within 8 hours of the commencement of) any analytical sequence.

9.2.2 Unless otherwise specified in the method, all samples, LCS's, MB's, MS's, Dup's must be bracketed by CCV's. Any CCV that is run in the middle of a sequence (between reportable runs) is called a "Mid-sequence CCV".

A Mid-Sequence CCV is used to:

9.2.3 Evaluate whether the *prior* samples can be reported: A sample is considered reportable unless the analyst judges the instrument to be unable to meet sensitivity requirements or identifies a system change resulting in irreproducible results.

9.2.4 Evaluate whether the *following* samples can be reported: An analyte can be reported only if the value (calculated concentration or calibration factor) for *that analyte* in the Mid-Sequence CCV is within ± 25% of the known value or calibration factor in the initial calibration, depending of which calibration method is used. If the samples *contain* target analytes for which the ± 25% was not met, the samples should be rerun. A "Non Detect" finding for any analyte *is reportable* unless the analyst judges the instrument to be unable to meet sensitivity requirements

9.2.5 Evaluate the chromatogram of any CCV that did not pass criteria carryover from a highly contaminated sample. Repeat the analysis of the CCV which failed if a non-instrument related problem caused the CCV to exceed the criteria. If the new CCV is acceptable, then reinject all samples with possible hits which followed the failed CCV in the sequence. If the results still do not meet criteria, generate a new calibration curve.

If a problem related to the GC system has been determined to be the cause of the failed CCV, perform whatever maintenance is necessary before injecting a CCV or recalibrating and proceeding with sample analysis.

9.2.6 Retention Time Windows

The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of 72 hours. Three times the standard deviation of a retention time is used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

Use the mid-level standards (CCVs) interspersed throughout the analysis sequence to evaluate the qualitative performance of the GC system. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reinjecting a CCV or recalibrating and proceeding with sample analysis.

9.2.7 Identification of Analytes

Identify a sample component by comparison of its retention time to the retention time of the daily standard chromatogram.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. A tentatively identified compound is confirmed when the retention time for the compound on the confirmatory detector is within the retention time window on that system. Confirmation is routinely done using GCMS techniques.

10. QA/QC REQUIREMENTS

10.1 QC Samples Required

For each analytical batch (up to 20 samples), method blank (MB), matrix spike (MS), duplicate matrix spike (DMS), and laboratory control sample (LCS) must be analyzed. The method blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

10.2 Acceptance Criteria

10.2.1 Acceptance criteria for initial and continuing calibration verification are given in Section 9.1 and 9.2 respectively.

10.2.2 Section 9.3 defines the acceptance criteria for the retention times of continuing calibration check standards.

11. CALCULATIONS

Sample results are calculated by the external standard method. If there are no interferences, results may also be calculated by the internal standard method, using Pe_3SnCl as the internal standard.

Aqueous Samples:

$$Conentration\ (\mu g / L) = \frac{(Cex)\ (vf)\ (D)}{Vs} \times C$$

Where Cex = Concentration in extract in µg/ml
VF = Final volume of extract in ml
D = Dilution factor
Vs = Volume of sample extracted, liters
C = Appropriate chloride-to-cation correction factor (see Table 2)

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D) \times 1000}{(W) \times 1000} \times C$$

Cex = Concentration in extract in µg/ml
VF = Final volume of extract in ml
D = Dilution factor
W = Weight of sample extracted. The wet or dry weight may be used, depending upon the specific client requirements.
C = Appropriate chloride-to-cation correction factor (see Table 2)

12. REFERENCES

12.1 *U.S. EPA Test Methods for Evaluating Solid Waste*, SW-846, September 1986, Revision 0.

12.2 Unger, M.A.; MacIntyre, W.G. Greaves, J.; Huggett, R.J., *GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexane Derivatives with Mass Spectrometric Confirmation, Chemosphere*, 1986, 16 (4): 461-470.

12.3 Krone, C.A.; Brown, D.W.; Burrows, D.G.; Bogar, R.G.; Chan, S.; Varanasi, U., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November, 1988.

TABLE 1

BUTYLTINS

	--Method Reporting Limits--		
Compound	Water(µg/L)	Soil(µg/Kg)	Tissue(µg/Kg)
Tetrabutyltin	0.1	3	10
Tributyltin	0.1	3	10
Dibutyltin	0.1	3	10
Monobutyltin	0.1	3	10

TABLE 2

CONVERSION FACTORS

Compound	Salt Cation	Cation - Sn
Tetrabutyltin	1	0.3419
Tributyltin	0.8911	0.4092
Dibutyltin	0.7666	0.5095
Monobutyltin	0.6230	0.6751

STANDARD OPERATING PROCEDURE

BUTYLTINS

SOC-BUTYL
Revision 3
September 1, 1999

Approved By:

Supervisor	9/1/99 Date
QA Manager	9-1-99 Date
Laboratory Manager	9-1-99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

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Will Not Be Updated

Standard Operating Procedure

for

BUTYLTINS

1. SCOPE AND APPLICATION

This procedure is used to determine the concentrations of butyltins in water, sediment and tissue. Tributyltin chloride (Bu_3SnCl) as well as butyltin trichloride ($BuSnCl_3$) dibutyltin dichloride (Bu_2SnCl_2), and tetrabutyltin (Bu_4Sn) can be determined by this method. The butyltin chloride salts of tributyltin, dibutyltin and monobutyltin are reported as butyltin cations. The current method reporting limit for each of these analytes is listed in Table 1.

2. METHOD SUMMARY

This procedure is based on techniques described in various papers (Unger, et. al; Krone, et.al.) for performing the extraction, derivitization, and analysis of mono-, di-, tri-, and tetrabutyltin chloride, with use of certain modifications. This procedure involves a 0.1% tropolone in methylene chloride extraction of the analytes of interest from an acidified sample, followed by a Grignard reaction of the hexane extract with Hexylmagnesium bromide (HxMgBr). The extract is then eluted through silica and alumina cartridge columns for cleanup of soil extracts, or Florisil columns for cleanup of tissue extracts, then analyzed by GC/FPD with a 610nm bandwidth filter. The extraction procedure used is outlined in the CAS SOP SOC-OSWT.

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount as established during initial calibration. The verification is done by analyzing for target analytes in standards in an appropriate solvent. ICV solutions are made from neat standards whose source is different from that used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and

analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard (Calibration) Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the ongoing accuracy of the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2. Since this procedure is often applied to "trace level" analyses, particular attention should be paid to ensuring that standards are of acceptable quality and purity.

4.3. Sulfur compounds cause the most direct interference with this determination. Copper shavings or powder is used to help remove the sulfur that might otherwise interfere with the flame photometric detector.

5. SAFETY

The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. Where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4°C from time of collection until extraction. Water samples should be extracted within 7 days and soil/sediment samples should be extracted within 14 days of collection. Tissue samples should be extracted within 1 year when stored frozen (-18°C) until extraction. Studies have not been done to determine sample or extract stability, however extracts should be analyzed within 40 days after extraction.

7. STANDARDS AND REAGENTS

7.1. Neat standards may be purchased from Aldrich, Alfa, or Acros. Chemical Abstract Service (C.A.S.) numbers may be used to identify the specific standards. Refer to the list below.

Standard	C.A.S. Number
$BuSnCl_3$	1118-46-3
Bu_2SnCl_2	683-18-1
Bu_3SnCl	1461-22-9
Bu_4Sn	1461-25-2
Pe_3SnCl	3342-67-4
Pr_3SnCl	2279-76-7
Tricyclohexyltin	3091-32-5

7.2. Stock Solutions - 5000 µg/mL

7.2.1. Butyltin Stock

A stock solution of Tetrabutyltin, Tributyltin, Dibutyltin, and Monobutyltin cations is prepared by weighing out the following amounts of the neat standards:

Tetrabutyltin	50.0 mg
Tributyltin chloride	56.1 mg
Dibutyltin dichloride	65.2 mg
Butyltin trichloride	80.2 mg

Dilute to 10 mL in DCM. Replace this solution yearly, or sooner if there are signs of degradation. Alternatively, certified stock solutions may be purchased (Restek) at 2000ppm in DCM.

7.2.2. Surrogate Stock

Stock solutions of each surrogate (tripropyltin chloride, tripentyltin chloride, or tricyclohexyltin chloride) are prepared together or individually by weighing 50 mg of the neat standards and diluting to 10 mL in DCM. These solutions are good for 1 year.

7.2.3. Independent Calibration Verification Stock Standard - Prepare a separate butyltin stock standard as described in 7.2.1 above, but using neat standards from a different source or use purchased stock solutions of a different source.

7.3. Working Standards - 100 µg/mL

7.3.1. Intermediate Butyltin Standard - Appropriately dilute the 5000 µg/mL stock solution for butyltins in Acetone (5000 µg/mL x 200 µL/10 mL of acetone). Replace every 2 months.

7.3.2. Intermediate Surrogate Standard - Appropriately dilute each 5000 µg/mL stock solutions of tripentyltin chloride and tripropyltin chloride into one mixture in acetone (5000 µg/mL x 200 µL/10 mL of acetone). Replace every 2 months.

7.3.3. Butyltin Spike Standard - Prepare a 5 ug/mL solution by diluting the intermediate butyltin standard in acetone (100 µg/mL x 2.50 mL/50 mL). Replace every 2 months.

7.3.4. Butyltin Surrogate Spike - Prepare a 5 ug/mL solution by diluting the intermediate butyltin standard in acetone (100 µg/mL x 2.50 mL/50 mL). Replace every 2 months.

7.4. Calibration Standards

7.4.1. The calibration stock standard is prepared by diluting 0.5 mL each of the working surrogate solution and the working spike solution to 10 µg/mL in 5 mL Hexane (100 µg/mL x 0.5 mL/5 mL) and derivitizing with Grignard reagent. The calibration stock standard is brought to 5mL final volume following alumina and silica gel cleanups. This standard can be prepared at the same time that a sample set is derivatized.

7.4.2. Calibration Standards

Calibration standards are prepared by diluting the calibration stock solution into appropriate levels. A minimum of three levels (five is recommended) is used to demonstrate linearity using a linear regression calibration fit, or by the using the average response factor. Recommended concentrations are: 5, 10, 50, 100, 500, 1000, and 2000 µg/L as cations. Each standard will include the surrogates. To convert the butyltin cation concentration to the corresponding chloride salt concentration, divide the cation conc. by the following correction factors:

Tributyltin	=	0.8910
Dibutyltin	=	0.7665
Monobutyltin	=	0.6230

7.5. ICV Standard Solution - Prepare a 100 ug/mL by diluting the 2000 ug/mL ICV stock in acetone (2000 ug/mL x 500uL/10mL in acetone). This solution should be replaced every 2 months. Add 200uL of this solution to 5mL of hexane and derivatize the resulting solution with Grignard reagent. Perform alumina and silica gel cleanups and bring to 200mL final volume. The resulting solution is good for 1 year.

7.6. Store all standards (neat and in solution) in a refrigerator of freezer (<4°C).

8. EQUIPMENT AND OPERATING CONDITIONS

8.1. Gas Chromatograph (GC) equipped with a flame photometric detector (FPD) and autosampler, HP 5890 with Enviroquant. A 610nm center wavelength, 10nm bandwidth filter for the FPD is required (purchased from Oriel, Stratford, Conn., Part No. 53295). The recommended GC columns and operating conditions are as follows:

8.1.1. Columns:

Restek RTX-5 30m x 0.32mm I.D., 0.5 µm film thickness (Cat. No. 10255) or equivalent.

Restek RTX-1701 megabore 30m x 0.53mm I.D., 1.0 µm film thickness.

8.1.2. GC conditions:

Flow settings:

Carrier flow (column)	40 cm/sec.
Makeup flow (detector)	15 ml/min.
Hydrogen flow (detector)	175 ml/min.
Air flow (detector)	100 ml/min.

Temperature program (both columns):

Temp 1	80°C
Time 1	1 minute
Rate 1	30°C/minute
Temp 2	170°C
Time 2	0 minutes
Rate 2	15°C/minute
Temp 3	250°C
Time 3	0 minutes
Rate 3	35°C/minute
Temp 4	340°C
Time 4	1.1 minutes

8.2. Data system - A computer system must be interfaced to the GC. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC data file and plot such response versus time. The data system must be capable of performing calibrations and quantitation calculations. HP Enviroquant is the current software in use.

8.3. Analytical balance (0.0001 g).

8.4. Volumetric flasks, syringes, vials, and bottles for standards preparation.

9. PREVENTATIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. The autosampler should be cleaned periodically. This includes turret cleaning and cleaning or replacing the syringe. Refer to manufacturer's instructions for autosampler restarting.

9.5. The detector should be cleaned and serviced as specified by the manufacturer.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training

program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Initial Calibration

Calibrate the GC immediately prior to conducting any analyses, using the external standard technique. Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area) versus the concentration in the standard. The ratio of the response to the amount injected, defined as the calibration factor (CF), is calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than or equal to 20% over the working range, linearity through the origin can be assumed, and the average calibration factor may be used in place of a calibration curve. Other options for establishing the calibration are given below. The analyst should evaluate the chromatographic baseline to ascertain if instability prevents accurate quantitation of low calibration standards.

If %RSD exceeds 20%, the analyst may elect to plot a linear regression curve. Print the plot and evaluate the calibration curve by reprocessing the low standard (at or near the MRL) to check for gross error. Curves should not be forced through zero.

The following should be used as warning limits:

- linear regression R^2 value < 0.990
- % RSD on the average RF's exceed 50%

If any of the above warning limits are exceeded, the analyst should do the following:

- Assess instrument's ability to attain MRL for this analyte. There needs to be sufficient sensitivity.
- Recalculate low, high and CCV points using new curve. The value calculated should be within 50% of true value on the low point, 15% on the CCV and 30% on the high point. If this is not the case, the curve needs to be rerun for that analyte.
- Obtain a secondary opinion from a senior analyst or supervisor.

11.2. Following initial calibration, verify the calibration by analyzing the ICV standard. Acceptance criteria for the ICV is 70-130% of the true value. However, values exceeding ± 20 %D should be scrutinized for possible dilution error. Analyze the ICV after each dilution and derivitization of the calibration stock standards. It is also recommended to analyze the ICV after each calibration curve is established, even if the same calibration standards have previously been verified.

11.3. Calibration Verification

11.3.1. The start of any sequence must include a CCV checked against the Initial Calibration (curve or average calibration factors). For any analyte to "pass", the response (calculated concentration or calibration factor) must be within ±25%D of the expected response. For this specific "passing" analyte, this CCV is valid for 24 hours and is considered an "Opening CCV". An Opening CCV must be run prior to (within 8 hours of the commencement of) any analytical sequence.

11.3.2. All samples, LCSs, MBs, MSs, Dups must be bracketed by CCVs. Any CCV that is run in the middle of a sequence (between reportable runs) is called a "Mid-sequence CCV". For any analyte to "pass", the response (calculated concentration or calibration factor) must be within ±25%D of the expected response when compared to the initial calibration. If the criteria is exceeded, determine necessary corrective action as follows:

11.3.2.1. Evaluate the chromatogram of any CCV that did not pass criteria. Repeat the analysis of the CCV which failed if a non-instrument related problem caused the CCV to exceed the criteria. If a problem related to the GC system has been determined to be the cause of the failed CCV, perform whatever maintenance is necessary before injecting a CCV or recalibrating and proceeding with further analyses. If the new CCV is acceptable, then reinject samples associated with the failing CCV as follows:

11.3.2.1.1. If the %D exceeded the upper limit of the criteria (>125%), the prior samples can be reported if the results are less the MRL. Samples with analytes detected above the MRL must be re-analyzed under passing CCV conditions.

11.3.2.1.2. If the %D was less than the lower limit of the criteria (<75%), take appropriate corrective action (such as evaluating standards, GC operation, etc.) to re-establish a valid calibration. This may include performing a new initial calibration. Re-analyze any samples run prior to the failing CCV.

11.3.2.2. Any samples following a failing CCV must be rerun.

11.4. Retention Time Windows

11.4.1. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of 72 hours. Three times the standard deviation of a retention time is used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.4.2. Use the mid-level standards (CCVs) interspersed throughout the analysis sequence to evaluate the qualitative performance of the GC system. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reinjecting a CCV or recalibrating and proceeding with sample analysis.

11.5. Sample Analysis

11.5.1. Following calibration analyses, analyze samples in a set referred to as an analytical sequence. The sequence includes field samples and QC samples bracketed by CCVs. Using the data system, setup the data acquisition to acquire data for each analysis into a distinct datafile. Refer to the SOP for Analytical Batches and Analytical Sequences for guidance on setting up analyses.

11.5.2. Identification of Analytes

11.5.2.1. Identify a sample component by comparison of its retention time to the retention time of the daily standard chromatogram.

11.5.2.2. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. A tentatively identified compound is confirmed when the retention time for the compound on the confirmatory detector is within the retention time window on that system. Confirmation is routinely done using secondary column and/or GCMS techniques.

12. QA/QC REQUIREMENTS

12.1. QC Samples Required

12.1.1. For each analytical batch (up to 20 samples), method blank (MB), matrix spike (MS), duplicate matrix spike (DMS), and laboratory control sample (LCS) must be analyzed.

The method blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

12.2. Acceptance Criteria

12.2.1. The ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples, or whenever significant changes to the procedures have been made. To do this, four reagent water samples are spiked with each target analyte, extracted, and analyzed. Calculate the average percent recovery and standard deviation. Since no specific criteria are defined for this analysis, the section supervisor/manager should review this data for reasonableness.

12.3. Method Detection Limits

12.3.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP for The Determination of Method Detection Limits.

12.3.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually for each sample matrix.

12.4. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Analytical Batches and Analytical Sequences. Routinely, these include:

12.4.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank.

12.4.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Appendix A. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken.

12.4.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

The acceptance limits for the MS/DMS are given in Appendix A. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

12.4.4. The acceptance limits for the surrogates are given in Appendix A. If surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be identified.

12.5. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION AND REPORTING

13.1. Calculations

Sample results are calculated by the external standard method. If there are no interferences, results may also be calculated by the internal standard method, using Pe_3SnCl as the internal standard, if the associated internal calibration is established. For external standard, use the following equations, for internal standard, use techniques described in EPA Method 8000B for quantification.

Aqueous Samples:

$$Conentration\ (\mu g / L) = \frac{(Cex)\ (vf)\ (D)}{Vs} \times C$$

Where
- Cex = Concentration in extract in µg/ml
- VF = Final volume of extract in ml
- D = Dilution factor
- Vs = Volume of sample extracted, liters
- C = Appropriate chloride-to-cation correction factor (see Table 2)

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D) \times 1000}{(W) \times 1000} \times C$$

- Cex = Concentration in extract in µg/ml
- VF = Final volume of extract in ml
- D = Dilution factor
- W = Weight of sample extracted. The wet or dry weight may be used, depending upon the specific client requirements.
- C = Appropriate chloride-to-cation correction factor (see Table 2)

13.2. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.3. Reporting

13.3.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.3.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.3.3. Sample concentrations are reported when all QC criteria for the analysis has been met or the results are qualified with an appropriate footnote.

14. TRAINING OUTLINE

The following items provide guidelines for training analysts.

- Review applicable literature (method references, etc.) and this SOP. Review the MSDS for all chemicals used in the analysis.
- Observe the procedure as performed by an experienced analyst at least three times.
- Assist in the procedure under the guidance of an experienced analyst for at least one month, preferably three months. During this training period, the analyst is expected to progress from a role of assisting to a role of performing the procedure with minimal oversight. Following this training period, the analyst is expected to complete an Initial Precision and Recovery (IPR) study as described in Section 12.1 for both solid and water matrices. Documentation of the IPR study should be forwarded to the analyst's training file.

15. REFERENCES

15.1. *U.S. EPA Test Methods for Evaluating Solid Waste*, SW-846, Method 8000B, Revision 2, December 1996.

15.2. Unger, M.A.; MacIntyre, W.G. Greaves, J.; Huggett, R.J., *GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexane Derivatives with Mass Spectrometric Confirmation, Chemosphere*, 1986, 16 (4): 461-470.

15.3. Krone, C.A.; Brown, D.W.; Burrows, D.G.; Bogar, R.G.; Chan, S.; Varanasi, U., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November, 1988.

TABLE 1

BUTYLTINS METHOD REPORTING LIMITS

	--Method Reporting Limits--			
Compound	Water(µg/L)	Porewater (ug/L)	Soil(µg/Kg)	Tissue(µg/Kg)
Tetrabutyltin	0.05	0.05	3	10
Tributyltin	0.02	0.02	1	2
Dibutyltin	0.05	0.05	1	2
Monobutyltin	0.05	0.05	1	2

TABLE 2

CONVERSION FACTORS

Compound	Salt Cation	Cation - Sn
Tetrabutyltin	1	0.3419
Tributyltin	0.8911	0.4092
Dibutyltin	0.7666	0.5095
Monobutyltin	0.6230	0.6751

APPENDIX A

QC ACCEPTANCE CRITERIA

SURROGATE SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compounds	Water	Soil/Sediment	Tissue
Tripropyltin	21-107	18-125	13-176
Tripentyltin	21-116	28-122	32-167

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Compound	Water	Soil/Sediment	Tissue
Tributyltin	23-131	27-162	17-185
Monobutyltin	17-128	8-161**	17-185*
Dibutyltin	16-118	8-161	17-185*
Tetrabutyltin	23-131*	27-162*	17-185*

MATRIX SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Matrix Spike Compound	Water	Soil/Sediment	Tissue
Tributyltin	20-116	13-163	10-206
Monobutyltin	20-116*	8-144**	20-153
Dibutyltin	20-116*	8-144	18-179
Tetrabutyltin	20-116*	13-163*	10-206*

* Advisory limits from Tributyltin should be used until sufficient historical data available to generate individual limits.

* * Advisory limits from Dibutyltin should be used until sufficient historical data available to generate individual limits.

STANDARD OPERATING PROCEDURE

EXTRACTION OF ORGANOTINS IN SEDIMENT, WATER, AND TISSUE MATRICES

SOC-OSWT
Revision 2

July 21, 1998

Approved By:

Supervisor	7/21/98 Date
QA Coordinator	7-21-98 Date
Laboratory Manager	7/21/98 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: LW for J.G.H. Date: 6-24-99
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

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EXTRACTION OF ORGANOTINS IN SEDIMENT, WATER, AND TISSUE MATRICES

1. SCOPE AND APPLICATION

This procedure is used to extract selected butyltins from sediments, water and tissues. The procedure is a preparative step for determination of butyltins by gas chromatography (SOP SOC-BUTYL). The procedure can also be applied to porewater samples.

2. SUMMARY OF METHOD

Butyltin compounds are extracted with an organic solvent using the technique suitable for the sample matrix. Sediment and tissue samples are extracted by tumbling. Water samples are extracted by separatory funnel. Extracts are then derivitized to their hexyl form using hexylmagnesiumbromide. After derivitization, extracts from sediment and water samples are cleaned up with silica and alumina; tissue sample extracts are cleaned up with Florisil. Extracts are then taken to final volume and analyzed by GC/FPD.

3. DEFINITIONS

Refer to the determinative procedure SOP (SOC-BUTYL) for applicable definitions.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running blanks.

4.2. In sediments samples, sulfur compounds cause the most direct interference with this procedure. Extracts are centrifuged and decanted to eliminate precipitated sulfur. Copper powder may also be used to help remove the sulfur that might otherwise interfere with the flame photometric detector.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses.

CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Appropriate precautions must be taken when preparing, handling, and storing Grignard reagent.

5.2. All samples should be treated as a potential hazard. Appropriate personal protective equipment must be worn when performing the procedure.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

Refer to the determinative procedure SOP (SOC-BUTYL).

7. APPARATUS AND MATERIALS

7.1. Materials used in sediment, tissue and water extractions.

7.1.1. pH paper, 0 - 14 range

7.1.2. 16x150mm and 25x150mm disposable glass culture tubes with Teflon lined screw caps

7.1.3. 0.5 ml, 1ml, 2 ml, 5 ml, and 10 ml serological pipettes

7.1.4. Pastuer pipets

7.1.5. 2 ml glass vials with Teflon lined crimp-top caps

7.1.6. Nitrogen evaporator

7.1.7. Centrifuge - capable of handling glassware in 7.1.2.

7.1.8. Vacuum pump and manifold

7.2. Materials for extraction of sediments

7.2.1. 2 L Teflon bottles with Teflon screw caps

7.2.2. Tumbler, capable of holding twelve 2 L Teflon bottles

7.2.3. Modified vacuum filtration funnel

7.2.4. Whatman No. 41 filter paper

7.2.5. Rotary evaporator with temperature controlled water bath

7.2.6. Glass round bottom flasks for rotary evaporator

7.2.7. Polypropylene funnels

7.2.8. Scoopulas

7.2.9. 400 ml beakers

7.3. Materials for extraction of waters

7.3.1. 500 ml, 1 L, and 2 L glass or Teflon separatory funnels with Teflon stopcock

7.3.2. 1 L glass fleaker

7.3.3. Vortex for culture tubes

7.3.4. 500 ml glass wide-mouth Erlenmeyer flasks

7.3.5. Sonic bath

7.3.6. Graduated cylinders (various sizes 100 - 1000 ml).

7.4. Materials for extraction of tissues

7.4.1. Vortex for VOA vials

7.4.2. Tumbler for VOA vials

7.4.3. 40 ml and 60 ml VOA vials with Teflon lined septa and screw caps

7.5. Materials for Grignard preparation

7.5.1. 500 ml round bottom three necked flask with glass stoppers

7.5.2. 30 cm condenser

7.5.3. 125 ml addition funnel

7.5.4. Two drying tubes filled with drierite and plugged with glass wool

7.5.5. Glass rod

7.5.6. Hot water bath

7.5.7. 100 ml graduated cylinders

7.5.8. 150 ml glass beaker

8. REAGENTS

8.1. Solvents: Dichloromethane (DCM), hexane, pentane, and anhydrous diethyl ether (J.T. Baker).

8.2. Tropolone in dichloromethane (DCM) - 0.1% (w/v) solution is prepared by adding 1 g tropolone per liter of DCM

8.3. Tropolone in hexane - 0.2% (w/v) solution is prepared by adding 2 g tropolone per liter of hexane

8.4. Reagent water (tap water).

8.5. D.I. Water

8.6. Sodium sulfate, purified by heating at 400°C for 4 hours

8.7. Concentrated HCl.

8.8. Fisher PrepSepR 1 g silica cartridges

8.9. Fisher PrepSepR 1 g alumina cartridges

8.10. Magnesium turnings (from Fisher).

8.11. Bromohexane (from Fluka).

8.12. Florisil cartridges (Supelco).

8.13. Surrogate and spiking solutions--see SOP SOC-BUTYL) Section 7.

8.14. Hexylmagnesiumbromide - CH3(CH2)5MgBr (Grignard). Prepare as follows:

8.14.1. All glassware used in Grignard preparation is baked at least 1 hour at $\geq$ 100°C to remove water. Glassware is assembled while still warm.

8.14.2. Add 14 g of magnesium to the three necked flask during assembly. The condenser is placed in the middle neck of the flask with a drying tube at the top of the condenser. A glass stopper is put in one neck of the flask, with the 125 ml addition funnel in the last flask opening. The addition funnel contains 40 ml bromohexane in 100 ml anhydrous ether, and is topped with a drying tube.

8.14.3. Add 2 ml bromohexane and 10 ml ether to the flask. A glass rod is used to crush 2 or 3 Mg chips. A hot water bath may be used to help reaction start. After reaction has started, hot water bath is removed.

8.14.4. Once reaction is started, the bromohexane solution in the addition funnel is added at a rate of about 3 drops per second. Once half of the bromohexane in ether solution has been added, another 140 ml of the solution is prepared in the addition funnel and added as the first portion was. Once all bromohexane solution has been added, the hot water bath is returned and reaction is refluxed for at least one hour. Grignard solution is now ready to use.

8.14.5. The reagent is may last up to 2 months after preparation. Due to the reactive nature of Grignard reagent, it should be checked for reactiveness prior to continued use. After 2 months, test weekly prior to use. A derivatization blank, a 1 ppm standard, and a 10 ppm standard are typically prepared ahead of sample derivatization to assure the purity and reactiveness of each batch of Grignard.

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

10. PROCEDURE

10.1. Extraction of Organotins From Sediments

10.1.1. Mix sample thoroughly and weigh approximately 50 g into a beaker. Record weight to nearest 0.01g. Method blank and laboratory control sample are made from an equal amount of sodium sulfate that has been rinsed with 0.1% tropolone/DCM solution. Mix samples with adequate tropolone/DCM rinsed sodium sulfate to dry sample. Add sample to a 2 L Teflon bottle and add appropriate surrogate and spike. Acidify with 2.5 ml concentrated HCl. Check pH to ensure pH <2. Add more acid if necessary. Add enough 0.1% tropolone/DCM solution to cover sample with at least 1/2" of solvent (approximately 250 ml). Cap tube, shake and vent to release pressure. Place bottles on tumbler, and tumble for at least 16 hours.

10.1.2. Pour sample into modified funnel with Whatman No. 41 filter paper, and filter by vacuum into a 1L round bottom flask. Evaporate the extract on the rotary evaporator to approximately 5 ml. Add 20 ml hexane and continue roto-vap to solvent exchange to hexane. Split each extract between two 16x150 mm culture tubes. Archive one split portion and complete the preparation on the other portion.

10.1.3. Using nitrogen, blow down the extract to approximately 3 ml. Add 2 ml D.I. water and vortex. Centrifuge to separate precipitates. Using a Pastuer pipet and pentane, quantitatively transfer the extract (solvent) layer to a 2nd culture tube. Evaporate extract down to approximately 2 ml.

10.1.4. Add 2 ml of Grignard reagent and vortex every 5 minutes over a 30 minute period. Add concentrated HCL slowly, vortexing as needed, until there is no reaction and the hexane layer is clear.

10.1.5. Using the vacuum manifold, set up alumina/silica cartridges and condition with 5 ml hexane. Discard hexane. Place samples in cartridges and bring down to almost dry, and elute with 6 ml of pentane, allowing this to go dry. Take extracts to a 0.5 ml final volume in pentane, and place in a 2 ml vial.

10.2. Extraction of Organotins In Water

10.2.1. Transfer 1 L of sample to a 2 L separatory funnel. Use tap water for method blank and laboratory control sample. Add surrogate and matrix spikes. Adjust to pH <2 with concentrated HCl. Add 30 ml of 0.2% tropolone in hexane solution. Shake funnel for 1 to 2 minutes with periodic venting. Repeat extraction two more times. Dry and evaporate extract down to approximately 5 ml in a 16x150 mm culture tube.

Note: For porewater samples, the available sample volume is typically limited to 500 ml or less. Determine the amount of sample available and adjust the volume extracted accordingly. Adjust the amount of reagents and surrogate and matrix spike amounts accordingly.

10.2.2. Add 2 ml Grignard, and vortex every 5 minutes over a 30 minute period. Add concentrated HCl slowly, vortexing as needed, until there is no reaction and hexane layer is clear.

10.2.3. Clean up using alumina and silica cartridge as described in section 10.1.5 and take to 1 ml (nominal) final volume.

10.3. Extraction of Organotins In Tissue

10.3.1. Mix sample thoroughly and weigh approximately 5 g into a 40 ml VOA vial. Add surrogate and matrix spikes. Acidify with 10 ml of 15 M HCl. Add 20 ml of 0.1% tropolone/DCM. Vortex 2 minutes, venting to release pressure. Place test tubes on tumbler, and tumble for at least 3 hours. Centrifuge extract. Remove 10 ml of tropolone/DCM layer and evaporate to 5 ml on the N-evap. Add 10 ml of hexane, evaporate to 2 ml, add 10 ml more hexane, evaporate to 2 ml again and take to 5 ml in hexane.

10.3.2. Continue with Grignard derivitization (10.1.4) and take to final volume as in section 10.1.5, using a Florisil cartridge instead of Alumina and silica gel cartridges.

11. QUALITY CONTROL

11.1. The QC samples required for the extraction batch are described in the Butyltins SOP (SOC-BUTYL) and the SOP for Analytical Batches and Analytical Sequences. Any method blanks or laboratory control samples should be subjected to exactly the same procedures as those used in actual samples.

11.2. Follow the applicable quality control guidelines outlined in Butyltins SOP (SOC-BUTYL).

12. DATA REDUCTION AND REPORTING

Use the appropriate benchsheet to record all sample preparation information. The benchsheet must be completed in full and reviewed by a 2nd analyst or supervisor. Sample preparation information recorded on the benchsheet is used to perform data reduction following GC analysis.

13. REFERENCES

13.1. Unger, M.A.; MacIntyre, W.G. Greaves, J.; Huggett, R.J., *GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexyl Derivatives with Mass Spectrometric Confirmation, Chemosphere*, 1986, 15 (4): 461-470

13.2. Krone, C.A.; Brown, D.W.; Burrows, D.G.; Bogar, R.G.: Chan, S.: Varanasi, U., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November, 1988.

STANDARD OPERATING PROCEDURE

EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE

SOC-OSWT

Revision 1

May 9, 1996

Approved By:

Teena Jones	Supervisor	5/9/96 Date
	QA Coordinator	5-9-96 Date
	Laboratory Manager	5-9-96 Date

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EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE

1. SCOPE AND APPLICATION

1.1 This method is used to extract butyltins from sediments, water and tissues.

2. SUMMARY OF METHOD

2.1 Butyltin compounds are extracted with an organic solvent. Sediment and tissue samples are extracted by tumbling. Water samples are extracted by separatory funnel. Extracts are then derivitized to their hexyl form using hexylmagnesiumbromide. After derivitization, samples are cleaned up with silica and alumina. They are then taken to final volume and analyzed by GC/FPD

3. INTERFERENCES

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running blanks.

3.2 Sulfur compounds cause the most direct interference with this determination. Copper powder is used to help remove the sulfur that might otherwise interfere with the flame photometric detector.

4. APPARATUS AND MATERIALS

4.1 Materials used in sediment, tissue and water extractions.

4.1.1 pH paper, 0 - 14 range

4.1.2 40 ml glass culture tubes with Teflon lined screw caps

4.1.3 1 ml, 5 ml, and 10 ml serological pipettes

4.1.4 2 ml glass vials with Teflon lined crimp-top caps

4.1.5 Nitrogen evaporator

4.2 Materials for extraction of sediments

4.2.1 2 L Teflon bottle with Teflon screw cap

4.2.2 Tumbler, capable of holding twelve 2 L Teflon tubes

4.2.3 Modified vacuum filtration funnel

4.2.4 Whatman No. 41 filter paper

4.2.5 Vacuum pump

4.2.6 Rotary evaporator with temperature controlled water bath

4.2.7 Glass round bottom flasks for rotary evaporator

4.2.8 Stir plates with Teflon coated magnetic stir bars

4.2.9 400 ml beakers

4.3 Materials for extraction of waters

4.3.1 2 L glass separatory funnels with Teflon stopcock

4.3.2 1 L glass fleaker

4.3.3 Vortex for culture tubes

4.4 Materials for extraction of tissues

4.4.1 Vortex for culture tubes

4.4.2 Tumbler for culture tubes

4.4.3 Centrifuge for culture tubes

4.5 Materials for Grignard preparation

4.5.1 250 ml round bottom three necked flask

4.5.2 30 cm condenser

4.5.3 125 ml addition funnel

4.5.4 Two drying tubes filled with drierite and plugged with glass wool

4.5.5 Glass rod

4.5.6 Hot water bath

4.5.7 Glass stopper for three necked flask

5. REAGENTS

5.1 Dichloroemthane (DCM)

5.2 Tropolone in dichloromethane (DCM) - 0.1% (w/v) solution is prepared by adding 1 g tropolone per liter of DCM

5.3 Hexane

5.4 Tropolone in hexane - 0.2% (w/v) solution is prepared by adding 2 g tropolone per liter of hexane

5.5 Reagent water

5.6 Sodium sulfate, purified by heating at 400°C for 4 hours

5.7 Concentrated HCl.

5.8 Fisher PrepSepR 1 g silica cartridge

5.9 Fisher PrepSepR 1 g alumina cartridge

5.10 Hexylmagnesiumbromide - $CH_3(CH_2)_5MgBr$ (Grignard) purchased from Aldrich or made in lab (See section 6)

5.11 Surrogate and spiking solutions--see SOP Butyltins (SOC-BUTYL) section 7.

5.12 Reagents used in Grignard preparation

5.12.1 3.5 g magnesium turnings from Fisher

5.12.2 50 ml anhydrous diethyl ether from Fisher

5.12.3 20 ml bromohexane from Aldrich

6. GRIGNARD PREPARATION

6.1 All glassware used in Grignard preparation is baked at least 1 hour to remove water. Glassware is assembled while still warm.

6.2 5 g of magnesium is added to the three necked flask during assembly. The condenser is placed in the middle neck of the flask with a drying tube at the top of the condenser. A glass stopper is put in one neck of the flask, with the 125 ml addition funnel in the last flask opening. The addition funnel contains 18 ml bromohexane in 40 ml anhydrous ether, and is topped with a drying tube.

6.3 2 ml bromohexane and 10 ml ether are added to the flask. A glass rod is used to crush 2 or 3 Mg chips. A hot water bath may be used to help reaction start. After reaction has started, hot water bath is removed.

6.4 Once reaction is started, the bromohexane solution in the addition funnel is added at a rate of about 3 drops per second. Once all bromohexane solution is added, the hot water bath is returned and reaction is refluxed for at least one hour. Grignard solution is now ready to use.

6.5 Due to the reactive nature of Grignard reagent, it should be prepared as close as possible to the time it is to be used to derivatize the organotin extracts. A derivatization blank and a 20 ppm standard are routinely prepared ahead of sample derivatization to assure the purity and reactiveness of each batch of Grignard.

7. PROCEDURE

7.1 Extraction of Organotins From Sediments

7.1.1 Mix sample thoroughly and weigh approximately 50 g into a beaker. Record weight to nearest 0.01g. Method blank and laboratory control sample are made from an equal amount of sodium sulfate that has been rinsed with 0.1% tropolone/DCM solution. Acidify with 2.5 ml concentrated HCl. Check pH to ensure pH <2. Add more acid if necessary. Mix samples with adequate tropolone/DCM rinsed sodium sulfate to dry sample. Add sample to a 2 L Teflon bottle and add appropriate surrogate and spike. Add enough 0.1% tropolone/DCM solution to cover sample with at least 1/2" of solvent (approximately 250 ml). Add 20 g of HCl rinsed copper powder. Cap tube, shake and vent to release pressure. Place bottles on tumbler, and tumble for at least 16 hours.

7.1.2 Pour sample into modified funnel with Whatman No. 41 filter paper, and filter by vacuum into a 500 ml round bottom flask. Evaporate the extract on the rotary evaporator to approximately 5 ml. Add 20 ml hexane and continue roto-vap to solvent exchange to hexane.

7.1.3 Set extracts on stir plates, and add 2 ml of Grignard. Let stir for 30 minutes. Then add 10 ml HCl, and continue stirring until hexane layer is clear. Pour contents of flask into a 40 ml test tube. Pull off hexane layer and blow down under nitrogen to less than 1 ml.

7.1.4 Using the vacuum manifold, set up alumina/silica cartridges and condition with 5 ml hexane. Discard hexane. Place samples in cartridges and bring down to almost dry, and elute with 3 ml of pentane, allowing this to go dry. Take extracts to a 0.5 ml final volume in pentane, and place in a 2 ml vial.

7.2 Extraction of Organotins From Water

7.2.1 Transfer 1 L of sample to a 2 L separatory funnel. Use tap water for method blank and laboratory control sample. Add surrogate and matrix spikes. Adjust to pH <2 with concentrated HCl. Add 20 ml of 0.2% tropolone in hexane solution. Shake funnel for 1 to 2 minutes with periodic venting. Repeat extraction two more times. Evaporate extract down to approximately 10 ml in a 40 ml culture tube.

7.2.2 Add 2 ml Grignard, and vortex every 5 minutes for 30 minutes. Add concentrated HCl slowly until there is no reaction and hexane layer is clear.

7.2.3 Clean up using alumina and silica cartridge as described in section 7.1.4 and take to 1 ml final volume.

7.3 Extraction of Organotins From Tissue

7.3.1 Mix sample thoroughly and weigh approximately 5 g into a 40 ml culture tube. Add surrogate and matrix spikes. Acidify with 10 ml of 15 M HCl. Add 20 ml of 0.1% tropolone/DCM. Vortex 2 minutes, venting to release pressure. Place test tubes on tumbler, and tumble for at least 3 hours. Centrifuge extract. Remove 10 ml of tropolone/DCM layer and evaporate to 5 ml on the N-evap. Add 10 ml of hexane, evaporate to 2 ml, add 10 ml more hexane, evaporate to 2 ml again and take to 5 ml in hexane.

7.3.2 Continue with Grignard derivitization and take to final volume as in section 7.1.4.

8. QUALITY CONTROL

8.1 Any method blanks or laboratory control samples should be subjected to exactly the same procedures as those used in actual samples.

8.2 Follow quality control guidelines outlined in Butyltins SOP.

9. REFERENCES

9.1 Unger, M.A.; MacIntyre, W.G. Greaves, J.; Huggett, R.J., *GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexane Derivatives with Mass Spectrometric Confirmation, Chemosphere*, 1986, 16 (4): 461-470

9.2 Krone, C.A.; Brown, D.W.; Burrows, D.G.; Bogar, R.G.: Chan, S.: Varanasi, U., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November, 1988.

STANDARD OPERATING PROCEDURE

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

SOC-8081
Revision 2
July 1, 1999

Approved By:

Supervisor	7-2-99 Date
QA Manager	7-2-99 Date
Laboratory Manager	7-2-99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
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DOCUMENT CONTROL

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ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Organochlorine Pesticides in water and soil/sediment using EPA Method 8081A. Table 1 indicates compounds that can be determined by this procedure and lists the method reporting limits (MRLs) in water and soil/sediment. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. Method detection limits which have been achieved are given in Table 1. A low-level sediment option may be used, with MRLs given in Table 2.

2. SUMMARY OF METHOD

2.1. This procedure provides gas chromatographic conditions for the detection of low concentration (typically parts-per-billion level organochlorine pesticides) pesticides. Target analytes are extracted from a sample using an appropriate sample extraction techniques and isolated via extract cleanup if needed. Liquid samples are extracted via separatory funnel or continuous liquid-liquid extraction. Soil/sediment samples are extracted using Soxhlet extraction, Ultrasonic extraction procedures, or Accelerated Solvent Extraction (Method 3545) .

2.2. A 1uL extract aliquot is analyzed using a gas chromatograph (GC) equipped with fused silica capillary columns and electron capture detectors (ECD). Identification is based on comparison of sample retention times to retention times for standard materials. Quantitative analysis is performed by using standards to produce a calibration curve, and using analyte response to determine the analyte extract concentration. Sample concentration is calculated using the extract concentration and the extracted sample weights, volumes and dilution factors.

2.3. The sensitivity of the procedure is usually depends on the level of interferences rather than instrument limitations. If interferences prevent the detection of analytes, GPC cleanup, Florisil cleanup, and sulfur cleanup may be used to reduce interferences. GPC cleanup is performed on all soil/sediment samples.

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

3.2. Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

3.3. Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

3.4. Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.5. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.6. Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.7. Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

3.8. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2. Interferences from phthalate esters introduced during sample handling can pose a problem with pesticide determinations. These can be removed using GPC cleanup. The

presence of elemental sulfur will result in peaks interfering with early eluting pesticides. Cleanup via method 3660 is used for the removal of sulfur if GPC cleanup is inadequate. Other co-extractables such as lipids, waxes, etc., can be removed via GPC cleanup.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses.

5.2. Follow normal CAS precautions as per the CAS Safety Manual. Sufficient care must be taken in handling solvents and standard solutions. Protective equipment should be used at all times. Protective equipment includes safety glasses, gloves and a lab coat. Consult the Material Safety Data Sheets (MSDS) or the Safety Officer for more information.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sampling should be performed according to SW-846 guidelines or project-specific procedures.

6.2. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction.

6.3. Water samples must be extracted within 7 days and the extracts analyzed within 40 days. Soil samples must be extracted within 14 days and the extract analyzed within 40 days.

7. STANDARDS, REAGENTS AND EQUIPMENT

7.1. Reagents

7.1.1. Stock Standard Solutions

Commercially prepared stock standards, certified by the manufacturer, are purchased from Ultra Scientific, Accustandard, and/or Absolute Standards.

7.1.2. Working Standard Solutions

7.1.2.1. Calibration standards are prepared that contain surrogates and analytes in hexane or iso-octane. The calibration standards are stored in the refrigerator for up to six months. The concentrations of the standards should encompass the range of expected concentrations of the samples to be analyzed. The nominal concentrations of the standards are 5 - 200 ppb for single component pesticides and 100-5000 ppb for Toxaphene.

7.1.2.2. Surrogate solution containing decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachlorometaxylene (TCMX), is prepared in acetone at 2 ppm. The surrogate solution is stored in the refrigerator for up to six months.

7.1.2.3. A matrix spike solution at 5 ppm is prepared by diluting the stock solution in acetone. This solution is stored in the refrigerator for up to six months. The matrix spike solution is added to all matrix spikes and lab control samples as outlined in section 10.

7.1.3. Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent

8. APPARATUS AND EQUIPMENT

8.1. GC Instrumentation (Equivalent may be used)

8.1.1. Gas Chromatograph, equipped with cool-on-column or split/splitless injection port that is temperature programmable with dual ECD, Hewlett Packard 5890. See Table 3 for typical chromatographic conditions. Helium is used as the carrier gas. Argon/methane mixture is used as the detector make-up gas.

8.1.2. Columns, Restek megabore columns typically are used;

Column 1 : Rtx-5 30m x 0.53mm ID, 1.0 µm df or equivalent*
Column 2 : Rtx-1701 30m x 0.53mm ID, 1.0 µm df or equivalent*
Guard Column: Rtx-5 5m x 0.53mm ID, 0.25 µm df, or equivalent

* Note: Column diameter and film thickness may vary depending on instrument. Refer to the instrument maintenance logbook for the columns used for a specific instrument configuration.

8.1.3. Autosampler, capable of reproducible 1-3 µL injections, Hewlett Packard 7673.

8.1.4. Data System - A computer data system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances. The most recent version of the manufacturer's software is preferred. HP Enviroquant.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8081/8081A, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

Water samples (1L) are extracted at a neutral, or as is, pH with methylene chloride, using either EPA Method 3510 or 3520. Soil/sediment samples are extracted using either EPA Method 3540, 3545, or 3550. A low-level sediment option may be used where the sample weight/volume are adjusted to lower the level of detection in the sample. Refer to the applicable extraction SOP.

11.2. Calibration

NOTE: Refer to the CAS protocols for organics analyses calibration (Attachment A). The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.2.1. Check for degradation of 4,4'-DDT and endrin by injecting a standard containing only 4,4'-DDT and endrin at 200 ppb.

$$\%\ Breakdown = \frac{Total\ DDT\ degratation\ peak\ area\ (DDE + DDD)}{Total\ DDT\ peak\ area\ (DDT + DDE + DDD)} \times 100$$

$$\%\ Breakdown = \frac{\begin{array}{c}Total\ endrin\ degratation\ peak\ area \\ endrin\ aldehyde + endrin\ Ketone\end{array}}{\begin{array}{c}Total\ endrin\ peak\ area \\ endrin + endrin\ aldehyde + endrin\ Ketone\end{array}} \times 100$$

If degradation of either DDT or endrin exceeds 15%, perform necessary maintenance before proceeding with calibration. The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each analytical sequence.

11.2.2. After determining that degradation is within acceptance, calibrate the system immediately prior to conducting any analyses. Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area or height) versus the concentration in the standard. For multi-component analytes, only those specified in the project plan or work specification are used for calibration.

Note: For Toxaphene and other multi-response components, a minimum of 3 peaks must be chosen for each compound. The peaks must be characteristic of the compound of interest.

11.2.3. The results can be used to determine a response factor (RF) for each compound. The average response factor (RF_a) is then calculated. The Relative Standard Deviation (RSD) must be less than 20% when average response factor is used. Alternatively, a linear curve with a correlation coefficient of 0.995 or greater may be used, or a quadratic calibration curve used.

11.2.4. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Calibration policy.

11.2.5. Continuing Calibration Verification

11.2.5.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A mid-level standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence.

11.2.5.2. The acceptance criteria for all analytes in the CCV analysis is a response (RF or concentration) within ± 15% D of the expected value, as compared to the initial calibration. Refer to the CAS protocols for organics analyses calibration for CCV evaluation protocols.

11.2.5.3. Use the mid-level standards interspersed throughout the analysis sequence to evaluate the qualitative performance of the GC system. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, the analysis sequence is ended. Perform whatever maintenance is necessary and inject another CCV. If the standard still falls outside of the daily retention time window, recalibrate and proceed with sample analysis. All samples that were injected after the sample exceeded the criteria must be reinjected.

11.2.6. Retention Time Windows

11.2.6.1. Establish retention time windows with the GC system in acceptable operating conditions. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000B for detailed instructions.

11.2.6.2. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

11.2.6.3. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.3. Sample Analysis

11.3.1. Table 2 indicates the typical operating conditions for the GC. Setup the analysis sequence of sample and QC samples. All samples, and QC samples must be bracketed by CCVs. Also, refer to the SOP for Analytical Batches and Analytical Sequences for guidance.

11.3.2. Calibrate the system as described in Section 11.2. Evaluate the CCVs as discussed in Section 11.2.2. In addition to calibration verification, use the CCVs interspersed throughout the analysis sequence to evaluate the qualitative performance of the GC system. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reinjecting a CCV or recalibrating and proceeding with sample analysis.

11.4. Identification of Analytes

11.4.1. Identify a sample component by comparison of its retention time to the retention time of the daily standard chromatogram.

11.4.2. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. A tentatively identified compound is confirmed when the retention time for the compound on the confirmatory detector is within the retention time window on that system.

11.4.3. Confirmation of all tentative hits should be made. Confirmation is made by injecting the sample extract on two columns with dissimilar phases simultaneously. If the retention time matches on both columns, then the hit for the analyte is considered a confirmed hit. Refer to the CAS policy for Confirmation of Organic Analytes.

11.4.4. For each multicomponent analyte, a minimum of 3 peaks are selected that are as unique to that analyte as possible. Quantitation in sample extracts is performed by comparing the average area of residue peaks to the average area of the same 3 peaks in the appropriate reference material.

11.5. Perform all necessary calculations as described in Sections 12 and 13.

12. QUALITY CONTROL

12.1. Initial Precision and Recovery Validation

The precision of the extraction procedure and the GC procedure must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with 1mL of the IPR solution, then extracting and proceeding with Section 11. The IPR solution can be purchased from a different vendor than the vendor used for calibration standards. The concentration of the analytes to be spiked may be found in method 3500B.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates (i.e. 1L of tap water for water MDLs, 15g sand for soil MDLs) with MDL spiking solution at a level below the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS SOP for The Determination of Method Detection Limits.

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Analytical Batches and Analytical Sequences. In general, these include:

12.3.1. A method blank is extracted and analyzed with every batch of 20 samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or reextraction and reanalysis.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 samples. The LCS is prepared by adding 50 µL of the matrix spike solution to tap water or 200 µL to sand, then proceeding with Section 9. For low level soil extraction, add 100 µL matrix spike solution.

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Table 4. If the lab control sample (LCS) fails acceptance limits for any of the compounds, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.3.3. A matrix spike (MS) and duplicate matrix spike (DMS) must be extracted and analyzed with every batch of 20 samples. The MS/DMS is prepared by adding the same volume of the matrix spike solution to the sample as listed for the LCS, then proceeding with Section 9. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = %recovery of the MS
R2 = %recovery of the DMS

The acceptance limits for the MS/DMS are given in Table 4. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.3.4. Surrogate spike is added to every sample, blank and spike prior to extraction. Two surrogate standards (tetrachloro-m-xylene and decachlorobiphenyl) are added to each sample; however, only one need be calculated for recovery. 100µL surrogate spike is added to waters, while 200-500µL is added to soils depending on the final volume. The surrogate is used to monitor extraction efficiency and retention time during GC analysis. Calculate surrogate percent recovery (%R) as:

$$\%R = S/V \times 100$$

Where S = The amount of surrogate recovered
V = The amount spiked/final volume

The acceptance limits for the surrogates are given in Table 4. If both surrogate recoveries are outside of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action include recalculation, reanalysis, or reextraction and reanalysis.

12.4. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Both detectors are used as primary and/or confirmatory systems when not interfered with by the sample matrix.

13.2. Calculations

13.2.1. Quantitation of analytes in sample extracts is performed by comparing total area of residue peaks to total area or peaks from the appropriate reference materials.

13.2.2. Quantitation of multi-response analytes:

13.2.2.1. The quantitation of Toxaphene and other multi-response analytes is accomplished by comparison of the sample chromatogram to that of the authentic standard. At the start of the analytical sequence the analyst should select which column is the primary quantitation column and which column is the secondary or confirmation column. All calibration acceptance criteria as described in section 11 must be met before reporting any results. The analyst must identify which column is the primary column by indication on the sequence run log. Sample results should then be reported off the primary column for that analytical sequence. Results may be reported from the secondary confirmation column if all calibration acceptance criteria as described in section 11are met.

13.2.2.2. Once the analyte pattern has been identified, compare the responses of 3-5 major peaks in the calibration standard with the peaks observed in the sample extract. The amount of analyte is calculated using the individual calibration factor for each of the 3-5 peaks and the calibration model selected in section 11. The concentration is determined using the 3-5 characteristic peaks and then the concentrations are averaged to determine the concentration. If there are interfering peaks with the 3-5 quantitation peaks that cause the average to be falsely overstated, then that interference peak is Q-deleted using the data system and the average is recalculated so that the average more truly represents the concentration in the sample.

13.2.3. The concentration of each analyte in the sample extract (Cex) is computed in µg/mL units using the calibration factor or calibration curve. The concentration of analytes in the original samples is computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample extracted, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where Cex = Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample extracted in grams. The wet or dry weight may be used, depending upon the specific client requirements.

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.4. Reporting

13.4.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.4.3. Sample concentrations are reported when all QC criteria for the analysis has been met or the results are qualified with an appropriate footnote.

14. TRAINING OUTLINE

14.1. The following items provide guidelines for training analysts.

14.1.1. Review applicable literature (method references, etc.) and this SOP. Review the MSDS for all chemicals used in the analysis.

14.1.2. Observe the procedure as performed by an experienced analyst at least three times.

14.1.3. Assist in the procedure under the guidance of an experienced analyst for at least one month, preferably three months. During this training period, the analyst is expected to progress from a role of assisting to a role of performing the procedure with minimal oversight. Following this training period, the analyst is expected to complete an Initial Precision and Recovery (IPR) study as described in Section 12.1 for both solid and water matrices. Documentation of the IPR study should be forwarded to the analyst's training file.

15. REFERENCES

15.1. EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 8081A, Revision 1

15.2. EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 8000B, Revision 2

TABLE 1

Organochlorine Pesticides
Target Analytes, Method Reporting Limits, and Method Detection Limits

Analyte	Water		Soil/sediment	
	MDL (ug/L)	MRL (ug/L)	MDL (mg/kg)	MRL (mg/kg)
Alpha-BHC	0.004	0.04	0.002	0.01
Gamma-BHC (Lindane)	0.004	0.04	0.001	0.01
Beta-BHC	0.005	0.04	0.0005	0.01
Heptachlor	0.003	0.04	0.0002	0.01
Delta-BHC	0.0007	0.04	0.002	0.01
Aldrin	0.001	0.04	0.0003	0.01
Heptachlor Epoxide	0.002	0.04	0.002	0.01
Endosulfan I	0.0006	0.04	0.0002	0.01
4,4'-DDE	0.002	0.04	0.0004	0.01
Dieldrin	0.002	0.04	0.0003	0.01
Endrin	0.002	0.04	0.0002	0.01
4,4'-DDD	0.002	0.04	0.0005	0.01
Endosulfan II	0.004	0.04	0.0003	0.01
4,4'-DDT	0.0006	0.04	0.0003	0.01
Endrin Aldehyde	0.0009	0.04	0.0006	0.01
Endosulfan Sulfate	0.001	0.04	0.0002	0.01
alpha-Chlordane	0.002	0.04	0.0003	0.01
gamma-Chlordane	0.0009	0.04	0.0003	0.01
Endrin Ketone	0.0009	0.04	0.0006	0.01
Methoxychlor	0.003	0.1	0.0005	0.02
Toxaphene	0.20	1	0.05	0.3

TABLE 2

Low Level Option for Organochlorine Pesticides in Soil and Sediment

Analyte	MRL (ug/kg)
Alpha-BHC	2
Gamma-BHC (Lindane)	2
Beta-BHC	2
Heptachlor	2
Delta-BHC	2
Aldrin	2
Heptachlor Epoxide	2
Endosulfan I	2
4,4'-DDE	2
Dieldrin	2
Endrin	2
4,4'-DDD	2
Endosulfan II	2
4,4'-DDT	2
Endrin Aldehyde	2
Endosulfan Sulfate	2
alpha-Chlordane	2
gamma-Chlordane	2
Endrin Ketone	2
Methoxychlor	4
Toxaphene	30

TABLE 3

Gas Chromatograph Operating Conditions

Gas Chromatograph:	HP5890 or equivalent
Injection Port Temperature:	83°C
Oven Temperature Program:	80°C to 185°C 35°C/min. hold 1 min. Ramp 4.5°C/min. to 280°C, hold 10 min.
Detector Temperature:	300°C
Injection Volume:	1-3 µL
Column 1:	30-m, 0.53 mm id, RTX-5 phase, 1.0 µ film thickness or equivalent
Column 2:	30-m, 0.53 mm id, RTX-1701 phase, 1.0 µ film thickness or equivalent.
Guard Column:	Rtx-1 1-5m-0.53mm id 1.5 µ or /5-m, 0.53 mm id, deactivated, uncoated fused silica column or equivalent.
Carrier Gas:	Helium
Auxillary Gas:	Argon/Methane
Data System:	HP Enviroquant

The above instrument temperatures may be modified when determining additional single response or multi-response pesticides.

TABLE 4

QC ACCEPTANCE CRITERIA

SURROGATE SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
Tetrachloro-m-xylene	19-107	16-104
Decachlorobiphenyl	15-122	23-116

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Compound	Water	Soil/Sediment
gamma-BHC (Lindane)	53-120	17-134
Heptachlor	16-117	24-118
Aldrin	13-113	26-120
Dieldrin	61-117	32-129
Endrin	61-124	34-131
4,4'-DDT	61-127	24-147

MATRIX SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Compound	Water	Soil/Sediment
gamma-BHC (Lindane)	38-131	23-120
Heptachlor	23-120	28-113
Aldrin	10-126	22-116
Dieldrin	26-139	32-126
Endrin	36-137	25-131
4,4'-DDT	17-140	14-151

Attachment A

CAS Organics Analysis Calibration Policy and Procedure

Columbia Analytical Services, Inc.
An Employee-Owned Company

MEMORANDUM

DATE: May 24, 1999

TO: Department Managers, Kelso Organics

FROM: Joe Wiegel

SUBJ: **INITIAL AND CONTINUING CALIBRATIONS FOR ORGANICS, DRAFT REVISION NO. 9**

This memo states our policy on performing and evaluating initial and continuing calibrations for organic analyses. The guidelines stated in the memo are designed as default specification. Unless stated in standard operating procedures and prescribed in the determinative method, this memo states our standard operating procedure for calibrations. This policy is consistent with EPA Method 8000B, SW-846, Third Edition, Update III.

1. General Calibration Guidelines

   1.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

   1.2. Calibrations for organic analyses must contain a minimum of five concentrations.

   1.3. The method reporting limit (MRL) must be supported by the calibration, typically as the low point in the calibration.

   1.4. The complete calibration (i.e., all initial calibration (ICAL) levels and the independent calibration verification (ICV) standard) must be analyzed prior to analysis of field or QC samples.

   1.5. A calibration may not be interrupted by a maintenance event.

   1.6. A calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated.

   1.7. Calibration points may be dropped at the endpoints of the curve as long as conditions 1.1, 1.2 and 1.3 are met.

1.8. Calibration points may not be dropped from the interior of a curve unless a catastrophic error (e.g., gross dilution error, missing internal standards, injection malfunction, etc.) is accounted for in a nonconformity and corrective action report (NCAR). In these circumstances, all the analytes in that calibration standard must be dropped from the calibration curve as corrective action.

1.9. Analysis of all field and QC samples must be preceded by an acceptable ICAL and ICV, or must be preceded by an acceptable CCV that verifies the ICAL.

2. Evaluation Guidelines for Initial Calibrations

2.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

2.2. Average response factor (RF*ave*) is the preferred calibration technique because linearity through the origin is assumed. As such, this technique allows the analyst to perform a more intuitive assessment of data below the lowest calibration standard. However, RF*ave* may not always be the best fit of calibration data. The analyst should use prior knowledge of the instrument, analyte response, and an assessment of the calibration data in determining whether RF*ave* is appropriate.

2.2.1. Acceptance criteria for RF*ave*:

GC and HPLC

2.2.1.1. Relative standard deviation (RSD) equal to or less than 20% for all compounds.

GC/MS

2.2.1.2. System performance and calibration check compounds (SPCC and CCC) must meet method criteria.

2.2.1.3. Relative standard deviation (RSD) equal to or less than 15% for all compounds.

Allowable Exceptions

2.2.1.4. Some analytes are recognized as marginal performing compounds. Typically, these are analytes that are not expected to meet the primary evaluation criteria due to the chemical nature of the compound. Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. The list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be indicated in the analytical SOP. Acceptance criteria for RF*ave* for these compounds are as follow:

GC and HPLC Procedures

2.2.1.4.1. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%

2.2.1.4.2. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 20%.

2.2.1.4.3. The RSD for marginal performing compounds must be less than or equal to 40%.

GC/MS Procedures

2.2.1.4.4. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

2.2.1.4.5. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.1.4.6. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 15%.

2.2.1.4.7. The RSD for marginal performing compounds must be less than or equal to 30%.

2.2.2. A calibration that has been processed using RF*ave* that does not meet the criteria described above may be used to report non-detected analytes. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. The following criteria apply:

2.2.2.1. For GC and HPLC procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%.

2.2.2.2. For GC/MS procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.2.3. Adequate sensitivity to detect and confirm the analyte at the MRL must be demonstrated by the lowest calibration standard.

2.2.2.4. All confirmed detections for analytes exceeding RSD criteria in the ICAL, regardless of concentration, must be reanalyzed to ensure accurate quantification following recalibration of the instrument.

2.3. When curves are used, least squares and quadratic fits are permissible. These fits will not be forced through the origin. As such, it may become impractical to report estimated concentrations in samples below the lowest standard of calibration. When used, the analyst must be trained on the significance of the Y-intercept when using curve fits. Specifically, the analyst must understand that very low responses may

quantify to false positives depending on where the curve intersects the Y axis. Acceptance criteria for these fits are as follow:

2.3.1. Least Squares: $R \geq 0.995$ or $R^2 \geq 0.990$

2.3.2. Quadratic: $COD \geq 0.990$

2.3.3. If a least squares fit is used, the curve must contain a minimum of five (5) calibration levels.

2.3.4. If a quadratic fit is used, the curve must contain a minimum of six (6) calibration levels and must be continuous along the function (i.e., it must consist of consecutive increasing or consecutive decreasing numerical values).

2.4. Because calibration curves can not be forced through the origin, the analyst should evaluate the effect the y-intercept will have on quantifying detections at or below the lowest standard of calibration. This evaluation should involve extrapolating the curve to a level of one half the lowest standard (using one half the area or area ratio). If the result is positive and less than the MRL, the curve may be used. If the result is equal to or less than zero, or if the result is equal to or greater than the MRL, the curve fit is not to be used.

2.5. The calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated. Acceptance criteria for all techniques (GC, HPLC and GC/MS) are as follow:

2.5.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

2.5.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

2.5.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±30% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

2.5.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. ICV acceptance criteria on these analytes is ±30% for GC and HPLC procedures and ±40% for GC/MS procedures.

3. Evaluation Criteria: Continuing Calibration Verification (CCV) Standards

3.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

3.2. For calibrations using RF*ave*, CCV Standards are evaluated based on % Difference (%Dif) of the response factor. %Dif is calculated as:

$$\%\,Dif = \frac{RFv - RFave}{RFave} \times 100$$

Where:

RFv is the response factor (also relative response factor) or calibration factor from the verification standard and ***RFave*** is the average response factor or calibration factor from the calibration.

3.3. For calibrations using Least Squares or Quadratic fits, CCV Standards are evaluated based on % Drift (%Drft) of the measured value compared to the expected value. %Drft is calculated as:

$$\%\,Drft = \frac{Measured\ concentration - Expected\ concentration}{Expected\ concentration} \times 100$$

3.4. CCV Acceptance Criteria:

GC and HPLC Procedures

3.4.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±15% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

3.4.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±30%.

GC/MS Procedures

3.4.5. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

3.4.6. The average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.7. For individual analytes, the maximum allowable %Dif or %Drft is ±20% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.8. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±40%.

3.5. Non-detected analytes can be reported from analytical sequences that contained CCVs that do not pass acceptance criteria. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. This includes reanalysis of QA/QC samples containing spiked amounts of target analyte. The following criteria apply:

3.5.1. For GC and HPLC procedures, the average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.2. For GC/MS procedures, the average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.3. The analysis must demonstrate adequate sensitivity to detect and confirm the analyte at the MRL. Therefore, all analytes being reported to the client can not exhibit a %Dif or %Drft greater than ±40%. For external calibrations, this criteria applies to both preceding and concluding CCVs.

3.5.4. All confirmed detections for analytes that fail acceptance criteria in the CCV, regardless of concentration, must be reanalyzed to ensure accurate quantification. This criteria applies to all samples and QA/QC samples analyzed in the sequence.

3.6. CCV standards must be analyzed at the start of each analytical sequence (except when the sequence is initiated with an ICAL and ICV).

3.7. Two sequential analyses of a CCV at the beginning of an analytical sequence can be performed in an effort to prime the instrument for analysis. Routine analysis of two or

more sequential CCV standards throughout the analytical sequence in an effort to ensure continuation of the sequence is not permitted.

3.8. CCV standards are analyzed at the following frequency:

External Standard Calibrations

3.8.1. CCV standards should be analyzed after every 10 injections of field and QC samples or every 12 hours, which ever is more frequent.

3.8.2. Samples with confirmed detections must be bracketted by acceptable CCV standards.

3.8.3. When a closing CCV standard is not acceptable, corrective action must be taken. A new CCV standard may be prepared and analyzed to demonstrate degradation of the standard as the cause of a CCV outlier. In this case, instument stability will be verified and samples analyzed prior to this CCV can be reported. CCV standards that are reinjected after minor instrument maintenance (e.g., injection port maintenance, column bake-out, installation of a new trap, etc.) do not verify instrument stability. Samples analyzed prior to this CCV must be reanalyzed.

Internal Standard Calibrations

3.8.4. The analysis sequence must be initiatied with an acceptable instrument tune (for GC/MS) and an acceptable CCV. The last injection or analysis in the sequence must be started within 12 hours from the time of sequence initiation.

3.8.5. Internal standard response in the CCV should be within 0.5 - 2 times the response observed in the mid point calibration standard in the curve. Failure to meet this criteria should prompt the analyst to check the internal standard solution for possible degradation or concentration, or may indicate the need for instrument maintenance.

3.8.6. Internal standard response in each sample must be within 0.5 - 2 times the response observed in the initial CCV standard of the analytical sequence (or the mid point calibration standard in the curve if the analytical sequence began with an ICAL). Perform reanalysis or dilutions to assess the effect of matrix interferences on internal standard responses.

Confirmation of Organic Analyte Identification and Quantification *

May 20, 1999

The following confirmation policy describes minimum standards that all laboratories must strive to meet. Specific contracts, programs, customers, analyses, etc. may require different confirmation procedures. Deviations from this policy must be documented so that sample analysis and data review will follow the appropriate procedures.

1. Confirmation is not necessary for MS analyses (Method 8000B, Section 7.9, second paragraph). However, mass spectral confirmation must meet the criteria stated in the applicable method. For example, in Methods 8260B and 8270C, Section 7.6 and in Method 524.2, Section 11.6. Regardless, at least two characteristic ions with acceptable relative intensities are necessary for a confirmed hit.

2. Confirmation is not necessary for multi-component analytes like gasoline, diesel, and other petroleum hydrocarbon products. However, a second column analysis is required for the multi-component pesticides (like technical chlordane, toxaphene, strobane, etc. (Method 8081A, Section 7.6)) and for PCB Aroclors (Method 8081A, Sections 1.6, 4.2, and 7.3 and Method 8082, Sections 1.6, 4.2, and 7.7).

3. Confirmation is not necessary for benzene, toluene, ethylbenzene, total xylenes, and naphthalene (BTEXN) from the PID analysis in the presence of a fuel pattern from the FID. Methyl *tert*-butyl ether (MTBE) must be confirmed, especially in the presence of gasoline because of interference from 1-pentene. Exception: the State of Arizona requires confirmation of BTEXN and MTBE even if gasoline is present in the sample. The analyst must document the presence of the fuel pattern on the PID raw data to justify not performing a confirmation analysis of BTEXN and MTBE.

4. Confirmation is not necessary if the composition of the sample matrix is well established by prior analyses and there is no information from the customer to suspect that the sample is different than the one which has been characterized (Method 8000B, Section 7.9, third paragraph).

5. Except for statements 3 and 4, all single-response GC and HPLC hits must be confirmed by one of the following techniques:

   - A second GC column with dissimilar stationary phase so that elution order and/or retention times are different (i.e., same detector but different columns);
   - Data from two dissimilar detectors, e.g., PID and FID, PID and ELCD, UV and fluorescence, etc. (i.e., same column but different detectors);
   - By mass spectrometry (MS), e.g., GC/MS; and
   - By HPLC UV data at two different wavelengths (although this may not be adequate for some analytes).

6. If two target analytes coelute in the initial calibration on two dissimilar columns, these columns cannot be used to report quantitative data for either analyte individually; however, the two columns can be used if the two analytes are always reported as an unresolved pair. Efforts should be taken to prevent this situation by selecting columns that adequately resolve the pair of analytes or by determining analysis parameters that will resolve the pair of analytes. When this situation can not be avoided, a third column may be required.

* Based on SW-846 Method 8000B, Sections 7.9 and 7.10.4 and U. S. EPA Technical Memorandum dated 8/7/98 (Clarification Regarding Use of SW-846 Methods). Additionally, U. S. EPA's 500-numbered methods refer to confirmation analyses but the descriptions are not as detailed as in Method 8000B.

8c. If the RPD is ≥ 100%:

An RPD ≥ 100% strongly suggests there is a chromatographic problem (such as a matrix interference), but if the problem is not apparent nor correctable, the MRL is to be elevated, the result reported as Not Detected with a flag, and the customer must be advised in the case narrative of the disparity between the two results. The MRL should be elevated to a level greater than the lower of the two results. To elevate the MRL, calculate the elevation factor as follows:

- Calculate the sample concentration of the analyte based on the lower of the two results;
- Raise this result by one unit of measurement;
- Divide the elevated result by the original MRL for the analyte; and
- Round up to the nearest positive integer the result of this calculation and raise the MRL by this factor.

Example:

Analyte	MRL	Lower Result (sample conc.)	Elevated Result	Elevation Factor (raw)	Elevation Factor (rounded)	Elevated MRL
Mirex	2.0 ppb	3.8 ppb	3.9 ppb	1.95	2	4.0 ppb

9. A GC or HPLC hit may be confirmed qualitatively by GC/MS.

9a. The GC/MS qualitative confirmation analysis may be done outside the tune window and is only to confirm peak identification.

9b. The GC or HPLC result is to be reported.

10. A GC or HPLC hit may be confirmed both qualitatively and quantitatively by GC/MS.

10a. If certification, contractual terms, project specifications, or program specifications requires the GC/MS confirmation analysis to be both qualitative and quantitative, then the GC/MS confirmation analysis must meet all the QC criteria established for the GC/MS analysis, such as within an acceptable tune window, following an acceptable CCV, etc.

10b. When a GC or HPLC hit is confirmed both qualitatively and quantitatively by GC/MS, the agreement between the quantitative results must be evaluated after the identification has been confirmed. The relative percent difference (RPD) between the two results is calculated using the following formula:

$$RPD = |R_1 - R_2| \div (R_1 + R_2)/2 \times 100$$

where R_1 is the GC or HPLC result and R_2 is the GC/MS result.

- If the RPD is ≤ 40%, and there is no evidence of chromatographic problems for the GC or HPLC analysis, report the higher result.

* Based on SW-846 Method 8000B, Sections 7.9 and 7.10.4 and U. S. EPA Technical Memorandum dated 8/7/98 (Clarification Regarding Use of SW-846 Methods). Additionally, U. S. EPA's 500-numbered methods refer to confirmation analyses but the descriptions are not as detailed as in Method 8000B.

STANDARD OPERATING PROCEDURE

PCBS AS AROCLORS

SOC-8082A
Revision 1
June 17, 1999

Approved By:

Julie Gish Supervisor	6/17/99 Date
QA Manager	6-17-99 Date
Laboratory Manager	6/16/99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

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PCBs as Aroclors - Method 8082

1. SCOPE AND APPLICATION

This procedure is used to determine the concentrations of PCBs as Aroclors using EPA Method 8082. This procedure is typically applied to water, sediment, and soil matrices but may also be applicable to tissue or various miscellaneous waste samples. Table 1 lists the analytes that can be determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) which have been achieved are given in Table 1.

2. METHOD SUMMARY

2.1. This procedure provides gas chromatographic conditions for the detection of parts-per-billion (ppb) levels of PCBs. The target PCBs are extracted from samples using the appropriate procedure for the sample matrix (see applicable SOP), analyzed, and reported as Aroclors. Prior to the use of this method, an appropriate sample extraction technique must be used to recover the analytes of interest. A 1 uL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by an electron capture detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard and by comparison of elution patterns to those of Aroclor standards. Quantitative analysis is performed by using the authentic standard to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2. The sensitivity of this method usually depends on the level of interferences rather than on instrument limitations. If interferences prevent detection of the analytes, GPC, florisil column cleanup, sulfur cleanup, or concentrated sulfuric acid cleanups are used to eliminate interferences in the analysis.

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

Initial (or Independent) Calibration Verification (ICV) - Initial calibration verification standards which are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. Once it is determined there is no systematic error in preparation of the calibration standards, they are considered valid for subsequent calibrations (as methods and expiration dates allow). The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of selected Aroclors are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Interferences by phthalate esters can pose a major problem in PCB determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large, late-eluting peaks, especially in the 15% and 50% fractions from the florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Phthalate contamination is not usually a problem in our laboratory operation.

4.2. The presence of elemental sulfur will result in interferences for most Aroclors. Cleanup via Method 3660 is used for the removal of sulfur. Other co-extractables such as lipids, waxes, etc., can be removed via GPC cleanup. Certain fractionization cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.

6.4. Water samples must be extracted within 7 days and the extracts analyzed within 40 days. Soil samples must be extracted within 14 days and the extract analyzed within 40 days. Extracts are stored under refrigeration until analysis.

7. APPARATUS AND EQUIPMENT

7.1. Gas Chromatograph (GC)

7.1.1. Analytical system complete with gas chromatograph suitable for splitless or on-column automated injection into a wide bore capillary column with an electron capture detector (ECD). Helium is used as the carrier gas; argon/methane mixture is used for the detector makeup gas (auxiliary gas).

7.1.2. GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

7.1.3. GC Columns

Column 1: RTX-5, 30-m fused silica column, or equivalent.
Column 2: RTX-1701, 30-m fused silica column, or equivalent.

Note: Column diameter and film thickness varies depending on the instrument. Refer to the instrument maintenance logbook for the column used for a specific instrument configuration.

7.1.4. Data System - A computer data system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances. The most recent version of the manufacturer's software is preferred (HP Chemstation/Enviroquant).

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent.

8.2. Stock Standard Solutions

8.2.1. Commercially prepared stock standards are purchased at a nominal 1000 ppm concentration. Alternatively, prepare stock standard solutions at a concentration of 1.00 µg/µL by dissolving 0.0100 g of assayed reference material in isooctane and diluting to volume in a 10 ml volumetric flask with solvent. When compound purity is assayed to be 96% or greater, the standard or neat chemical can be used without a correction for purity to calculate the concentration of the stock standard.

8.2.2. Transfer stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 ± 2°C and protect from light. Check stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards indicates a problem.

8.3. Calibration Standards:

8.3.1. Prepare calibration standards at a minimum of five concentration levels containing equal concentrations of both Aroclor 1016 and 1260 by dilution of the stock standard(s) with isooctane or hexane. One of the concentration levels should be at or below a concentration representing the sample method reporting limit (MRL). This should provide a concentration near, but above the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Standards from 0.025 to 5 ppm are routinely prepared for each Aroclor used to calibrate the GC/ECD.

8.3.2. Standards of the other five Aroclors are prepared for use as retention time/pattern standards and to establish calibration factors for these Aroclors. Dilute the stock standard(s) with isooctane or hexane to the appropriate concentration level (1ppm is routinely prepared for each Aroclor used to calibrate the GC/ECD).

8.3.3. Calibration standard solutions must be replaced after six months, or sooner, if comparison with check standards indicate a problem.

8.4. Surrogate Standards: Decachlorobiphenyl (DCB) is used as the surrogate. Prepare a 2 ug/mL solution of DCB in acetone. For calibration, the DCB calibration standards are usually prepared in conjunction with Aroclors 1016/1260 (section 8.3.1).

8.5. Matrix spike solution: Prepare a spiking solution at 40 ug/mL containing both Aroclor 1016 and 1260 by dilution of the stock standard(s) with acetone.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8082, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

Water samples are extracted at a neutral, or as is, pH with methylene chloride, using either EPA Method 3510C or 3520C. Sediment, soil, and solid samples are extracted using either EPA Method 3540C, 3545, or 3550B. A "low level" soil/sediment option may be used. Refer to the CAS SOPs for those procedures. Additional sample cleanup procedures may be employed as appropriate for the samples.

11.2. Calibration

NOTE: Refer to the CAS protocols for organics analyses calibration (Attachment A). The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.2.1. Prepare a minimum of 5 calibration standards containing equal concentrations of both Aroclor 1016 and 1260 by dilution of the stock standard(s) with isooctane or hexane. Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining five

Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors. The concentrations should correspond to the mid-point of the linear range of the detector.

A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

11.2.2. Calibrate the system immediately prior to conducting any analyses. Refer to Table 2 for instrument conditions. Starting with the standard of lowest concentration, analyze each 1016/1260 calibration standard and tabulate response (peak area) versus the concentration in the standard. Refer ot Table 2 for instrument conditions. Calculate the ratio of the response to the amount injected the (calibration factor) for each analyte at each standard concentration. For 1016/1260 and DCB, the Relative Standard Deviation (RSD) must be less than 20% when average response factor is used. Refer to the CAS Organics Calibration Policy for alternate calibration procedures.

11.2.3. Analyze each of the single-point calibration standards of the other 5 Aroclors. Calculate the calibration factor (CF) for each analyte at each standard concentration.

11.2.4. Each calibration of each Aroclor is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Calibration policy.

11.3. Calibration Verification

11.3.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A mid-level standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence.

11.3.2. The acceptance criteria for all analytes in the CCV analysis is a response (RF or concentration) within ± 15% D of the expected value, as compared to the initial calibration. Refer to the CAS protocols for organics analyses calibration for CCV evaluation protocols.

11.4. Retention Time Windows

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Analysts should consult Method 8000 for the details of establishing retention time windows.

11.5. Gas Chromatography

11.5.1. Set up an analytical sequence for the standards and samples to be analyzed. Calibrate the system as described in Section 11.2. Refer to Table 2 for typical instrument operating conditions. The same conditions must be used for samples as for calibration and QC analyses. Ensure that the instrument configuration is correct and that any necessary maintenance has been performed. Figure 1 shows a typical analysis sequence.

11.5.2. Evaluate the CCVs as indicated in Section 11.3. Use the mid-level standards interspersed throughout the sample analysis sequence to evaluate the qualitative performance of the GC system. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reanalyzing the CCV or recalibrating and proceeding with sample analysis. All samples that were injected after the sample exceeded the criteria must be reinjected if initial analysis indicated the presence of any analytes of interest.

FIGURE 1

Analysis Sequence

ICB	Initial Calibration Blank
Standard 1	1016/1260 ICAL
Standard 2	1016/1260 ICAL
Standard 3	1016/1260 ICAL
Standard 4	1016/1260 ICAL
Standard 5	1016/1260 ICAL
Standard 6	1221 midpoint
Standard 7	1232 midpoint
Standard 8	1242 midpoint
Standard 9	1248 midpoint
Standard 10	1254 midpoint
CCB	Continuing Calibration Blank
ICV	Independent Calibration Verification
Method Blank	
LCS	Laboratory Control Sample
Sample 1	
Sample 2	
Sample 3	
Sample 4	
Sample 5	
Sample 6	
Matrix Spike	
Duplicate Matrix Spike	
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
Sample 7	
Sample 8	
Sample 9	
Sample 10	
etc.	
etc.	
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification

Note: If a valid 1016/1260 initial calibration is in place, an new set of initial calibration standards may not be required. In this case, CCV standards are analyzed at the beginning of the sequences in place of initial calibration standards.

12. QA/QC REQUIREMENTS

12.1. Refer to Section 8.0 of Method 8082 for general QC protocol. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8082 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP for The Determination of Method Detecion Limits.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Analytical Batches and Analytical Sequences. The Quality Assurance Manual (Section 12) provides guidance for interpretation of QC results. Ongoing QC samples include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Appendix I. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

12.3.3.1. The acceptance limits for the MS/DMS are given in Appendix I. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

12.3.4. The acceptance limits for the surrogates are given in Appendix I. If surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be identified.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Identification of PCB's as Aroclors

Tentative identification of PCB's as Aroclors is made when the pattern of peaks in the sample chromatogram matches the pattern of peaks in the Aroclor standard itself. There also needs to be agreement between the retention times and response ratios of the 3-5 selected quantitation peaks in the sample chromatogram and the Aroclor standard. Each tentative identification must be confirmed using a second GC column of dissimilar phase.

Weathering of PCB's in the environment and changes resulting for waste treatment processes may alter the pattern of a specific Aroclor so it does not closely match an Aroclor standard. The earlier eluding peaks will often diminish in comparison to the later eluding peaks. Samples may also include mixtures of two or more Aroclors. For both of these reasons a high level of analyst expertise is required to interpret complex chromatograms.

13.2. Quantitation of PCB's as Aroclors:

13.2.1. The quantitation of PCB's as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard or standards. At the start of the analytical sequence the analyst should select which column is the primary quantitation column and which column is the secondary or confirmation column. All calibration acceptance criteria as described in section 11must be met before reporting any results. The analyst must identify which column is the primary column by indication on the sequence run log. Sample results should then be reported off the primary column for that analytical sequence. Results may be reported from the secondary confirmation column if all calibration acceptance criteria as described in section 11are met.

13.2.2. Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the calibration standard of that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 peaks and the calibration model selected in section 11. The concentration is determined using the 3 to 5 characteristic peaks and then the concentrations are averaged to determine the concentration of the Aroclor. If there are interfering peaks with the 3 to 5 quantitation peaks that cause Aroclor average to be falsely overstated, then that interference peak is Q-deleted using the data system and the average is recalculated so that the average more truly represents the concentration in the sample. This often occurs when there are more than one Aroclor in a sample extract or if pesticides are present. Quantitation of mixed Aroclors will require the selection of peaks which are not shared in common by both Aroclors.

13.2.3. Using the data system, calculate the concentration of each analyte in the sample extract (Cex) µg/ml units using the calibration factor or calibration curve (Section 11). The sample concentration computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in extract in µg/ml
Vf = Final volume of extract in ml
D = Dilution factor
Vs = Volume of sample extracted, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)\ x\ 1{,}000}{(W)\ x\ 1{,}000}$$

Where Cex = Concentration in extract in µg/ml
Vf = Final volume of extract in ml
D = Dilution factor
W = Weight of sample extracted. The wet or dry weight may be used, depending upon the specific client requirements.

13.2.4. Sample concentrations are reported when all QC criteria for the analysis has been met or the results are qualified with a footnote.

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.4. Reporting

13.4.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transfered to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14. REFERENCES

Polychlorinated Biphenyls (PCBs) as Aroclors, Method 8082, Revision 0, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

TABLE 1

Target Analytes, Method Reporting Limits, and Method Detection Limits

	Water		Soil/Sediment		Soil/Sediment (low level)	
Aroclor	MRL (ug/L)	MDL (ug/L)	MRL (mg/kg)	MDL (mg/kg)	MRL (ug/kg)	MDL (ug/kg)
Aroclor 1016	0.2	0.02	0.1	0.03	10	4
Aroclor 1221	0.2	0.09	0.1	0.03	10	-
Aroclor 1232	0.2	0.03	0.1	0.02	10	-
Aroclor 1242	0.2	0.04	0.1	0.03	10	-
Aroclor 1248	0.2	0.04	0.1	0.02	10	-
Aroclor 1254	0.2	0.03	0.1	0.01	10	-
Aroclor 1260	0.2	0.02	0.1	0.02	10	3

TABLE 2

Gas Chromatograph Operating Conditions

Gas Chromatograph:	Hewlett-Packard Model 5890 or equivalent w/ECD
Injection Port Temperature:	300°C
Oven Temperature Program:	150°C for one minute, 15°/min ramp to 280°C, hold 13 min.
Detector Temperature:	350°C
Injection Volume:	1µL
Column:	30 m, RTX-5 and 30 m RTX-1701. *
Carrier Gas:	Helium
Auxillary Gas:	Argon/Methane
Data System:	HP Enviroquant

* The instrument temperatures may be modified depending on the instrument used. Also, the GC column diameter and film thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analyses.

APPENDIX I

QC Acceptance Limits

SURROGATE SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
Decachlorobiphenyl	18-123	40-134

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Compound	Water	Soil/Sediment
Aroclor 1016	32-115	36-123
Aroclor 1260	35-132	40-136

MATRIX SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Matrix Spike Compound	Water	Soil/Sediment
Aroclor 1016	37-109	18-146
Aroclor 1260	42-129	23-146

Attachment A

CAS Organics Calibration Policy

Columbia Analytical Services, Inc.
An Employee-Owned Company

MEMORANDUM

DATE: May 19, 1999

TO: Department Managers, Kelso Organics

FROM: Joe Wiegel

SUBJ: **INITIAL AND CONTINUING CALIBRATIONS FOR ORGANICS, DRAFT REVISION NO. 9**

This memo states our policy on performing and evaluating initial and continuing calibrations for organic analyses. The guidelines stated in the memo are designed as default specification. Unless stated in standard operating procedures and prescribed in the determinative method, this memo states our standard operating procedure for calibrations. This policy is consistent with EPA Method 8000B, SW-846, Third Edition, Update III.

1. General Calibration Guidelines

   1.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

   1.2. Calibrations for organic analyses must contain a minimum of five concentrations.

   1.3. The method reporting limit (MRL) must be supported by the calibration, typically as the low point in the calibration.

   1.4. The complete calibration (i.e., all initial calibration (ICAL) levels and the independent calibration verification (ICV) standard) must be analyzed prior to analysis of field or QC samples.

   1.5. A calibration may not be interrupted by a maintenance event.

   1.6. A calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated.

   1.7. Calibration points may be dropped at the endpoints of the curve as long as conditions 1.1, 1.2 and 1.3 are met.

1.8. Calibration points may not be dropped from the interior of a curve unless a catastrophic error (e.g., gross dilution error, missing internal standards, injection malfunction, etc.) is accounted for in a nonconformity and corrective action report (NCAR). In these circumstances, all the analytes in that calibration standard must be dropped from the calibration curve as corrective action.

1.9. Analysis of all field and QC samples must be preceded by an acceptable ICAL and ICV, or must be preceded by an acceptable CCV that verifies the ICAL.

2. Evaluation Guidelines for Initial Calibrations

2.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

2.2. Average response factor (RF*ave*) is the preferred calibration technique because linearity through the origin is assumed. As such, this technique allows the analyst to perform a more intuitive assessment of data below the lowest calibration standard. However, RF*ave* may not always be the best fit of calibration data. The analyst should use prior knowledge of the instrument, analyte response, and an assessment of the calibration data in determining whether RF*ave* is appropriate.

2.2.1. Acceptance criteria for RF*ave*:

GC and HPLC

2.2.1.1. Relative standard deviation (RSD) equal to or less than 20% for all compounds.

GC/MS

2.2.1.2. System performance and calibration check compounds (SPCC and CCC) must meet method criteria.

2.2.1.3. Relative standard deviation (RSD) equal to or less than 15% for all compounds.

Allowable Exceptions

2.2.1.4. Some analytes are recognized as marginal performing compounds. Typically, these are analytes that are not expected to meet the primary evaluation criteria due to the chemical nature of the compound. Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. The list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be indicated in the analytical SOP. Acceptance criteria for RF*ave* for these compounds are as follow:

GC and HPLC Procedures

2.2.1.4.1. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%

2.2.1.4.2. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 20%.

2.2.1.4.3. The RSD for marginal performing compounds must be less than or equal to 40%.

GC/MS Procedures

2.2.1.4.4. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

2.2.1.4.5. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.1.4.6. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 15%.

2.2.1.4.7. The RSD for marginal performing compounds must be less than or equal to 30%.

2.2.2. A calibration that has been processed using RF*ave* that does not meet the criteria described above may be used to report non-detected analytes. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. The following criteria apply:

2.2.2.1. For GC and HPLC procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%.

2.2.2.2. For GC/MS procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.2.3. Adequate sensitivity to detect and confirm the analyte at the MRL must be demonstrated by the lowest calibration standard.

2.2.2.4. All confirmed detections for analytes exceeding RSD criteria in the ICAL, regardless of concentration, must be reanalyzed to ensure accurate quantification following recalibration of the instrument.

2.3. When curves are used, least squares and quadratic fits are permissible. These fits will not be forced through the origin. As such, it may become impractical to report estimated concentrations in samples below the lowest standard of calibration. When used, the analyst must be trained on the significance of the Y-intercept when using curve fits. Specifically, the analyst must understand that very low responses may

quantify to false positives depending on where the curve intersects the Y axis. Acceptance criteria for these fits are as follow:

2.3.1. Least Squares: $R \geq 0.995$ or $R^2 \geq 0.990$

2.3.2. Quadratic: $COD \geq 0.990$

2.3.3. If a least squares fit is used, the curve must contain a minimum of five (5) calibration levels.

2.3.4. If a quadratic fit is used, the curve must contain a minimum of six (6) calibration levels and must be continuous along the function (i.e., it must consist of consecutive increasing or consecutive decreasing numerical values).

2.4. Because calibration curves can not be forced through the origin, the analyst should evaluate the effect the y-intercept will have on quantifying detections at or below the lowest standard of calibration. This evaluation should involve extrapolating the curve to a level of one half the lowest standard (using one half the area or area ratio). If the result is positive and less than the MRL, the curve may be used. If the result is equal to or less than zero, or if the result is equal to or greater than the MRL, the curve fit is not to be used.

2.5. The calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated. Acceptance criteria for all techniques (GC, HPLC and GC/MS) are as follow:

2.5.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

2.5.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

2.5.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±30% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

2.5.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. ICV acceptance criteria on these analytes is ±30% for GC and HPLC procedures and ±40% for GC/MS procedures.

3. Evaluation Criteria: Continuing Calibration Verification (CCV) Standards

3.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

3.2. For calibrations using RF*ave*, CCV Standards are evaluated based on % Difference (%Dif) of the response factor. %Dif is calculated as:

$$\%Dif = \frac{RFv - RFave}{RFave} \times 100$$

Where:

RFv is the response factor (also relative response factor) or calibration factor from the verification standard and ***RFave*** is the average response factor or calibration factor from the calibration.

3.3. For calibrations using Least Squares or Quadratic fits, CCV Standards are evaluated based on % Drift (%Drft) of the measured value compared to the expected value. %Drft is calculated as:

$$\%Drft = \frac{\text{Measured concentration - Expected concentration}}{\text{Expected concentration}} \times 100$$

3.4. CCV Acceptance Criteria:

GC and HPLC Procedures

3.4.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±15% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

3.4.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±30%.

GC/MS Procedures

3.4.5. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

3.4.6. The average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.7. For individual analytes, the maximum allowable %Dif or %Drft is ±20% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.8. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±40%.

3.5. Non-detected analytes can be reported from analytical sequences that contained CCVs that do not pass acceptance criteria. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. This includes reanalysis of QA/QC samples containing spiked amounts of target analyte. The following criteria apply:

3.5.1. For GC and HPLC procedures, the average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.2. For GC/MS procedures, the average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.3. The analysis must demonstrate adequate sensitivity to detect and confirm the analyte at the MRL. Therefore, all analytes being reported to the client can not exhibit a %Dif or %Drft greater than ±40%. For external calibrations, this criteria applies to both preceding and concluding CCVs.

3.5.4. All confirmed detections for analytes that fail acceptance criteria in the CCV, regardless of concentration, must be reanalyzed to ensure accurate quantification. This criteria applies to all samples and QA/QC samples analyzed in the sequence.

3.6. CCV standards must be analyzed at the start of each analytical sequence (except when the sequence is initiated with an ICAL and ICV).

3.7. Two sequential analyses of a CCV at the beginning of an analytical sequence can be performed in an effort to prime the instrument for analysis. Routine analysis of two or

more sequential CCV standards throughout the analytical sequence in an effort to ensure continuation of the sequence is not permitted.

3.8. CCV standards are analyzed at the following frequency:

External Standard Calibrations

3.8.1. CCV standards should be analyzed after every 10 injections of field and QC samples or every 12 hours, which ever is more frequent.

3.8.2. Samples with confirmed detections must be bracketted by acceptable CCV standards.

3.8.3. When a closing CCV standard is not acceptable, corrective action must be taken. A new CCV standard may be prepared and analyzed to demonstrate degradation of the standard as the cause of a CCV outlier. In this case, instrument stability will be verified and samples analyzed prior to this CCV can be reported. CCV standards that are reinjected after minor instrument maintenance (e.g., injection port maintenance, column bake-out, installation of a new trap, etc.) do not verify instrument stability. Samples analyzed prior to this CCV must be reanalyzed.

Internal Standard Calibrations

3.8.4. The analysis sequence must be initiatied with an acceptable instrument tune (for GC/MS) and an acceptable CCV. The last injection or analysis in the sequence must be started within 12 hours from the time of sequence initiation.

3.8.5. Internal standard response in the CCV should be within 0.5 - 2 times the response observed in the mid point calibration standard in the curve. Failure to meet this criteria should prompt the analyst to check the internal standard solution for possible degradation or concentration, or may indicate the need for instrument maintenance.

3.8.6. Internal standard response in each sample must be within 0.5 - 2 times the response observed in the initial CCV standard of the analytical sequence (or the mid point calibration standard in the curve if the analytical sequence began with an ICAL). Perform reanalysis or dilutions to assess the effect of matrix interferences on internal standard responses.

STANDARD OPERATING PROCEDURE

CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION (GC/ECD)

SOC-8082C
Revision 1
June 10, 1999

Approved By:

Julie Fish — Supervisor — 6/17/99 Date

QA Manager — 6-17-99 Date

Laboratory Manager — 6/16/99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
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CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION (GC/ECD)

1. SCOPE AND APPLICATION

1.1. This procedure is used specifically to determine the concentrations of certain PCB congeners in water, soil and sediment using EPA method 8082. This procedure may also be applicable to various tissue samples. The procedure is intended to determine these compounds at trace levels and therefore is not applicable to miscellaneous waste matrices and oils. More appropriate methods are available for the determination of PCBs (as Aroclors) in waste matrices.

1.2. There are 209 identified congeners for which the method may possibly be applied. Table 1 lists the analytes that can routinely be determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) which have been achieved are given in Table 1.

2. METHOD SUMMARY

This procedure is based on EPA Method 8082. This procedure provides sample preparation techniques and GC/ECD conditions for the detection PCB congeners at trace levels. Samples are extracted using an appropriate technique for the sample matrix. Cleanup steps are used to remove unwanted co-extracted compounds and/or matrix interferences. Depending on the sample matrix, some cleanup steps may not be necessary. The final sample extract is analyzed by simultaneous dual column gas chromatography using an electron capture detector. The compounds are separated on fused silica capillary columns with differing chromatographic separation characteristics. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard. Quantitative analysis is performed by using the authentic standard to produce a response factor or calibration curve. The calibration data is used to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

1.9 This method should be used by analysts experienced in the use of inductively coupled plasma mass spectrometry (ICP-MS), the interpretation of spectral and matrix interferences and procedures for their correction. A minimum of six months experience with commercial instrumentation is recommended.

1.10 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must document and have on file the required initial demonstration performance data described in Section 9.2 prior to using the method for analysis.

2.0 SUMMARY OF METHOD

2.1 An aliquot of a well mixed, homogeneous aqueous or solid sample is accurately weighed or measured for sample processing. For total recoverable analysis of a solid or an aqueous sample containing undissolved material, analytes are first solubilized by gentle refluxing with nitric and hydrochloric acids. After cooling, the sample is made up to volume, is mixed and centrifuged or allowed to settle overnight prior to analysis. For the determination of dissolved analytes in a filtered aqueous sample aliquot, or for the "direct analysis" total recoverable determination of analytes in drinking water where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, and then diluted to a predetermined volume and mixed before analysis.

2.2 The method describes the multi-element determination of trace elements by ICP-MS.[1-3] Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are detected by an electron multiplier or Faraday detector and the ion information processed by a data handling system. Interferences relating to the technique (Sect. 4) must be recognized and corrected for. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standards.

3.0 DEFINITIONS

3.1 Calibration Blank - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument (Sect. 7.6.1).

METHOD 200.8

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES
BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for determination of dissolved elements in ground waters, surface waters and drinking water. It may also be used for determination of total recoverable element concentrations in these waters as well as wastewaters, sludges and soils samples. This method is applicable to the following elements:

Analyte		Chemical Abstract Services Registry Numbers (CASRN)
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Lead	(Pb)	7439-92-1
Manganese	(Mn)	7439-96-5
Mercury	(Hg)	7439-97-6
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Thallium	(Tl)	7440-28-0
Thorium	(Th)	7440-29-1
Uranium	(U)	7440-61-1
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

Estimated instrument detection limits (IDLs) for these elements are listed in Table 1. These are intended as a guide to instrumental limits typical of a system optimized for multielement determinations and employing commercial instrumentation and pneumatic nebulization sample introduction. However, actual method detection limits (MDLs) and linear working ranges will be dependent on the sample matrix, instrumentation and selected operating conditions. Given in Table 7 are typical MDLs for both total recoverable determinations by "direct analysis" and where sample digestion is employed.

Initial (or Independent) Calibration Verification (ICV) - Initial calibration verification standards which are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. Once it is determined there is no systematic error in preparation of the calibration standards, they are considered valid for subsequent calibrations (as methods and expiration dates allow). The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of selected Aroclors are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing equipment may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks. Specific selection of reagents and the use of solvents purified by distillation in all-glass systems may be required. Analysts should pay particular attention to the introduction of contamination from phthalate esters and use sample handling practices which minimize the introduction of the compounds into extracts.

4.2. Interferences coextracted from samples will vary considerably from source to source, depending upon the sample matrix. Cleanup techniques are generally required to eliminate significant levels of interferences. The presence of organochlorine pesticides in the sample can represent a potential interference which may exist after cleanup steps have been taken.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method may not be precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.

6.4. Water samples should be extracted within 7 days and the extracts analyzed within 40 days. Soil and sediment samples should be extracted within 14 days and the extract analyzed within 40 days.

7. APPARATUS AND EQUIPMENT

7.1. Gas Chromatograph System

7.1.1. An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless or on-column injection and all required accessories, including syringes, analytical columns, and gases.

7.1.2. Gas Chromatograph - Equipped with cool-on-column or split/splitless injection port that is temperature programmable with dual ECD, Hewlett Packard 5890. See Table 2 for typical chromatographic conditions.

7.1.3. GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

7.1.4. Columns : Column 1: 60 meter Restek RTX-5, 0.32mm i.d., 0.25 µm, or equivalent. Column 2: 60 meter Restek RTX-1701, 0.32mm i.d., 0.25 µm, or equivalent.

7.1.5. Data System - A computer system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that performs analysis of chromatographic data (response vs. time) and allows the user to program qualitative and quantitative parameters which result in analyte identification and quantitation. The most recent version of the manufacturer's software is preferred (HP Chemstation/Enviroquant).

7.2. Analytical Balance (0.0001 g), syringes, bottles, and vials as required. All bottles and vials should use Teflon-lined caps or septa.

7.3. Sample preparation apparatus - Glassware and associated apparatus as required to perform sample extractions as described in SOPs for methods 3510, 3540, and 3550. Gel-Permeation Chromatography cleanup apparatus as described in EPA Method 3640. Other cleanup apparatus as specified in EPA Methods 3630 and 3665.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Methylene chloride, hexane, acetone, methanol, or other solvents as appropriate. Pesticide quality or equivalent.

8.2. Stock Standard Solutions

8.2.1. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or verified against an independent source. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions of at least 10 µg/ml by dissolving an appropriate weight (nearest .0001 g) reference standard in hexane, isooctane, or methylene chloride and diluting to volume in a 10 ml volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be

96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards indicates a problem.

8.3. Intermediate Standard Solutions: Dilute the appropriate volume of the stock standard(s) to yield a nominal 1 ug/ml concentration. Dilute using hexane or isooctane.

8.4. Calibration Standards: Calibration standards at a minimum of five concentration levels for each parameter of interest are prepared through dilution of the intermediate standards with hexane or isooctane. One of the concentration levels should be at the method reporting limit (MRL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define a linear range. Calibration solutions must be monitored closely for signs of degradation. The recommended replacement interval is 6 months.

8.5. Surrogate Standard: The performance of the extraction, cleanup, and analytical system and the effectiveness of the method in dealing with each sample matrix is monitored by spiking each sample, method blank, and matrix spike with a surrogate compound. For this analysis, the surrogate selected is hexabromobiphenyl. Prepare a spiking solution at 4 ug/ml. Store at 4°C and protect from light. Surrogate spiking solutions are replaced periodically based on the stability of the compounds and frequency of use.

8.6. Matrix Spike Standards: Matrix spike solutions are prepared at a level representative of the calibration mid-range. Prepare a solution that contains all QC target analytes. Either a subset of the target list or the entire target list is reported depending on project requirements. Store at 4±2°C and protect from light. Matrix spike solutions are replaced periodically based on the stability of the compound(s) and/or frequency of use.

8.7. Internal Standard Solution: The internal standard used is tetrachloro-m-xylene. Prepare a working standard at a concentration of 10 ug/ml. Each extract undergoing analysis is spiked with 10µL of the internal standard solution, resulting in a concentration of 100 ng/ml.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8082, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Extraction

11.1.1. Water samples: Water samples are extracted using EPA Method 3510. Refer to CAS SOP for *Separatory Funnel Liquid-Liquid Extraction*. The extraction solvent used is methylene chloride and the nominal sample size is 1 liter. Extracts are brought to a volume of 1.0 ml. The extract cleanup is then performed using the silica gel and acid/permanganate cleanups described in section 11.2. The GPC cleanup is optional, depending on the level of coextracted interferences in the sample. Mercury cleanup to remove sulfur may also be necessary.

11.1.2. Soil/sediment samples: Soil and sediment samples are extracted using EPA Method 3550. Refer to CAS SOP for *Ultrasonic Extraction*. The extraction solvent is 1:1 methylene chloride/acetone. The nominal sample size is 30g. Extracts are brought to a volume of 10 ml. The extract cleanup is then performed using GPC, silica gel, and acid/permanganate. Mercury cleanup to remove sulfur may also be necessary.

11.1.3. Tissue Samples: Tissue samples are extracted using a soxhlet extraction procedure based on EPA Method 3540. Refer to CAS SOP for *Soxhlet Extraction*. The extraction solvent used is 2.5:1 methylene chloride/methanol. The nominal sample size is 20g. Extracts are brought to a volume of 10 ml. The extract cleanup is then performed using GPC, silica gel, and acid/permanganate. Mercury cleanup to remove sulfur may also be necessary.

11.2. Sample extract cleanup - refer to the flowchart in Figure 1 which outlines the cleanup procedure.

11.2.1. Cleanup procedures performed on the samples should follow the flowchart shown in Figure 1. For water samples the GPC and Mercury cleanups are optional, based on the appearance or expected level of interferences. For soil, sediment, and tissue samples, the flowchart should be followed.

11.2.2. Gel Permeation Chromatography - Perform the cleanup using the technique described in EPA Method 3640. Refer to CAS SOP for *Gel Permeation Chromatography*. A PCB-specific window is selected that allows for collection of the extract fraction containing the target compounds.

11.2.3. Silica Gel - Perform the cleanup using the cartridge cleanup technique described in EPA Method 3630. Cartridges containing 1 gram of silica gel are eluted with 5 ml of hexane to condition the cartridge. Follow this by adding 1 ml of the sample extract. Elute with 3 ml of hexane and collect.

11.2.4. Acid/permanganate - Perform the cleanup using the technique described in EPA Method 3665. This cleanup must be performed on all sample extracts.

11.2.5. Sulfur cleanup - If excessive amounts of sulfur are encountered in the sample extract, as determined by extract screening, the extract must go through additional cleanup to remove sulfur. Perform the cleanup using the technique described in the CAS SOP for *Sulfur Cleanup*.

11.3. Calibration

NOTE: Refer to the CAS protocols for organics analyses calibration (Attachment A). The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.3.1. The GC is configured to perform simultaneous analysis using the two columns and dual ECD detectors. The columns are cofigured using a splitter which diverts flow from the guard column to the two analytical columns. Operating conditions must be the same for all calibration analyses and related sample analyses. The recommended GC operating conditions are given in Table 2.

11.3.2. Initial Calibration

Calibration standards at a minimum of 5 concentration levels are analyzed. Analyze 1 µL of each calibration standard and tabulate the peak area and concentration for each compound. Calculate response factors (RFs) for each compound as follows:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

where:

Ax	=	Area of the compound being measured.
Ais	=	Area of the internal standard.
Cis	=	Concentration of the internal standard (ng/mL).
Cx	=	Concentration of the compound being measured (ng/mL).

The average RF is calculated for each compound. The percent relative standard deviation (%RSD) is calculated for each compound. If the RF value is constant over the calibration range (<20% RSD) the average RF is used for quantitation. Refer to the CAS Organics Calibration Policy for alternate calibration procedures. If the initial calibration criteria cannot be met, the standards and/or GC system should be inspected and corrective action taken. Injection port cleaning and/or removal of a portion of the column inlet may be required.

11.3.3. The initail calibration is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Calibration policy.

11.4. Retention Time Windows

11.4.1. Establish retention time windows with the GC system in acceptable operating conditions. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000B for detailed instructions.

11.4.2. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

11.4.3. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.5. Calibration Verification

11.5.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A mid-level standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence. If internal standard calibration is used, a CCV after samples is not required.

11.5.2. Compare the response factor with the average response factor from the initial calibration. The acceptance criteria for all analytes in the CCV analysis is a response (RF or concentration) within ± 15% D of the expected value, as compared to the initial calibration. If a calibration model other than average RF is used, compare the calculated result to the true value rather than using Rfs. Refer to the CAS Organics Calibration Policy for CCV evaluation protocols. Calculate the percent difference using the following:

$$\%\,Difference = \frac{\overline{RF_I} - RF_c}{\overline{RF_I}} \times 100$$

where:

RFi = Average response factor from initial calibration.
RFc = Response factor from current verification check standard.

11.6. Sample Analysis

11.6.1. Sample extracts are generally screened prior to analysis (see Figure 1). Dilution(s) may be made prior to analysis. Add the internal standard spiking solution to each extract to obtain the nominal extract concentration of 100 ng/ml. Analyze the extract using the recommended operating conditions specified in Table 2, ensuring that the same conditions are used for samples as for calibration.

11.6.2. Perform all qualitative and quantitative measurements as described in the following sections. Store the extracts at 4±2° C, protected from light in vials equipped with unpierced Teflon lined septa.

12. QA/QC REQUIREMENTS

12.1. Method Validation

12.1.1. Initial Precision and Recovery Validation

The precision of the extraction procedure and the GC procedure must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked at a mid-level concentration, then extracted, and analyzed. Currently no specific criteria are available from method references. However, precision of the 4 replicates should approximate what is typically found with semi-volatile analytes analyzed by GC/ECD. The average recovery should approximate what is typically seen with PCB analyses. Refer to EPA Method 8082 for single lab IPR data.

12.1.2. Method Detection Limits

A method detection limit (MDL) study must be undertaken for a given matrix before analysis of samples can begin. To establish low detection limits that are precise and accurate, the analyst must perform the MDL procedure for each matrix of interest. Spike a minimum of seven samples with MDL spiking solution at a level below, or near the expected MRL. Follow the procedures outlined in the CAS SOP for The Determination of Method Detection Limits.

12.2. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Analytical Batches and Analytical Sequences. The Quality Assurance Manual (Section 12) provides guidance for interpretation of QC results. Ongoing QC samples include:

12.2.1. Method Blank

A method blank is extracted and analyzed with every batch of 20 samples, or daily, whichever is more frequent, to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or reextraction and reanalysis.

12.2.2. Laboratory Control Sample

A laboratory control sample (LCS) must be extracted and analyzed with every batch of 20 samples, or daily, whichever is more frequent. The LCS is prepared by adding a

known amount of the matrix spike solution to a blank sample matrix, then proceeding preparation and analysis. Calculate the % recovery as follows:

$$\%R = X/TV \times 100$$

Where	X	=	Concentration of the analyte recovered
	TV	=	True value of amount spiked

The acceptance criteria are given in Appendix I. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.2.3. Matrix Spike

A matrix spike (MS) and duplicate matrix spike (DMS) must be extracted and analyzed with every batch of 20 samples. The MS/DMS is prepared by adding a known amount of the matrix spike solution to the sample then proceeding with preparation and analysis. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where	X	=	Concentration of the analyte recovered
	X1	=	Concentration of unspiked analyte
	TV	=	True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where	R1	=	%recovery of the MS
	R2	=	%recovery of the DMS

The acceptance limits for the MS/DMS are given in Appendix I. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.2.4. Surrogate

Surrogate spike is added to every sample, blank and spike prior to extraction. Add a known amount of the surrogate spike to all samples and QC samples in the extraction

set. The surrogate is used to monitor extraction efficiency. Calculate surrogate percent recovery (%R) as:

$$\%R = S/V \times 100$$

Where S = The amount of surrogate recovered
V = The amount spiked/final volume

The surrogate acceptance limits are given in Appendix I. If the surrogate recovery is outside of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action include recalculation, reanalysis, or reextraction and reanalysis.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis

13.1.1. The sample component RRT must compare within ± 0.06 RRT units of the RRT for the compound in the opening CCV.

13.1.2. Confirmation of all tentative hits must be made. Confirmation is made by injecting the sample extract on two columns with dissimilar phases simultaneously. If the retention time matches on both columns, then the hit for the analyte is considered a confirmed hit. As further confirmation, subsequent quantitations on the two columns should agree. Refer to the CAS Confirmation Policy for guideance. If on one column, known coeluting compounds exist, a quantitation difference may not be an appropriate measure of confimation. In the case of known coeluting compounds on one column, using quantitation on the two columns as a measure of confirmation should take into account the relative amounts of the two components. When *Aroclors* are known to be present, the quantitations on the two columns may not agree within 40%D and the analyst's judgement should be used to determine if the results represent a positive *congener* result at that quantitation level. Confirmation by GCMS may also be used if the concentration is high enough for detection and accurate spectral identification. A GCMS confirmation need only be qualitative in nature.

13.2. Quantitative Analysis

13.2.1. When a compound has been identified, the quantitation of the compound is based on the area of the compound identified. The quantitation technique used is the same as the calibration technique for the specific component.

13.2.2. Internal Standard quantitation.

Calculate the concentration of each identified analyte in the sample as follows:

Water: $$concentration\ (\mu g / L) = \frac{(A_x)(I_s)(V_t)}{(A)_{is}(RF)(V_o)(V_i)}$$

where:

Ax = Area of the compound being measured.
Is = Amount of internal standard injected (ng).
Vt = Volume of total extract, taking into account dilutions (i.e. a 1-to-10 dilution of a 1 ml extract will mean Vt = 10,000 µL).
Ais = Area of the internal standard.
RF = Average response factor for compound being measured.
Vo = Volume of water extracted (ml).
Vi = Volume of extract injected (µL).

Sediment/Soil Sludge (on a dry-weight basis) and Tissue (normally on a wet-weight basis):

$$concentration\ (\mu g / kg) = \frac{(Ax)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

where:

Ax, Is, Vt, Ais, RF, Vi = Same as for water.
Ws = Weight of sample extracted or diluted in grams.
D = % dry weight of sample/100, or 1 for a wet-weight basis.

13.2.3. Quadratic Curve quantitation:

13.2.3.1.1. Using the peak area for the identified component, use the calibration curve constructed during initial calibration to determine the solution (extract) concentration. Use the solution concentration to determine sample concentration as follows:

Water: $$Concentration\ (\mu g / L) = \frac{(Cex)(Vf)(D)}{(Vs)}$$

where:

Cex = Calculated concentration in the extract analyzed (ug/mL)
Vf = Final volume of extract (mL).
D = Dilution factor.
Vs = Volume of sample extracted (L).

Sediment/Soil Sludge (on a dry-weight basis) and tissue (normally on a wet-weight basis):

$$Concentration\ (ng / g) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

where:

Cex = Calculated concentration in the extract analyzed (ng/uL)
Vf = Final volume of extract (uL).
W = Weight of sample extracted or diluted in grams.
D = % dry weight of sample/100, or 1 for a wet-weight basis.

13.2.4. If the quantitation result is over the calibration range for any compound, a dilution re-analysis is required.

13.3. Data Review

Data for the analytical sequence is compiled in a data "pack" which includes all calibration, QC, and sample data for the sequence. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.4. Reporting

13.4.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transfered to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14. REFERENCES

Polychlorinated Biphenyls (PCBs) by Capillary Column Gas Chromatography, EPASW846, Test Methods For Evaluating Solid Waste, Update III, Method 8082, Revision 0, December, 1996.

Determinative Chromatographic Separations, EPASW846, Test Methods For Evaluating Solid Waste, Update III, Method 8000B, Revision 2, December, 1996.

Extraction Methods 3510C (Separatory Funnel Liquid-Liquid Extraction), 3520C (Continuous Liquid-Liquid Extraction), 3540C (Soxhlet Extraction), 3545 (Pressurized Fluid Extraction), and 3550B (Ultrasonic Extraction); EPASW846, Test Methods For Evaluating Solid Waste, Update III, December, 1996.

Cleanup Methods 3630C (Silica Gel Cleanup), 3640A (Gel-Permeation Cleanup), 3665A (Sulfuric Acid / Permanganate Cleanup); EPASW846, Test Methods For Evaluating Solid Waste, Updates II and III, September 1994 and December 1996.

FIGURE 1

CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS)

SAMPLE CLEANUP FLOWCHART

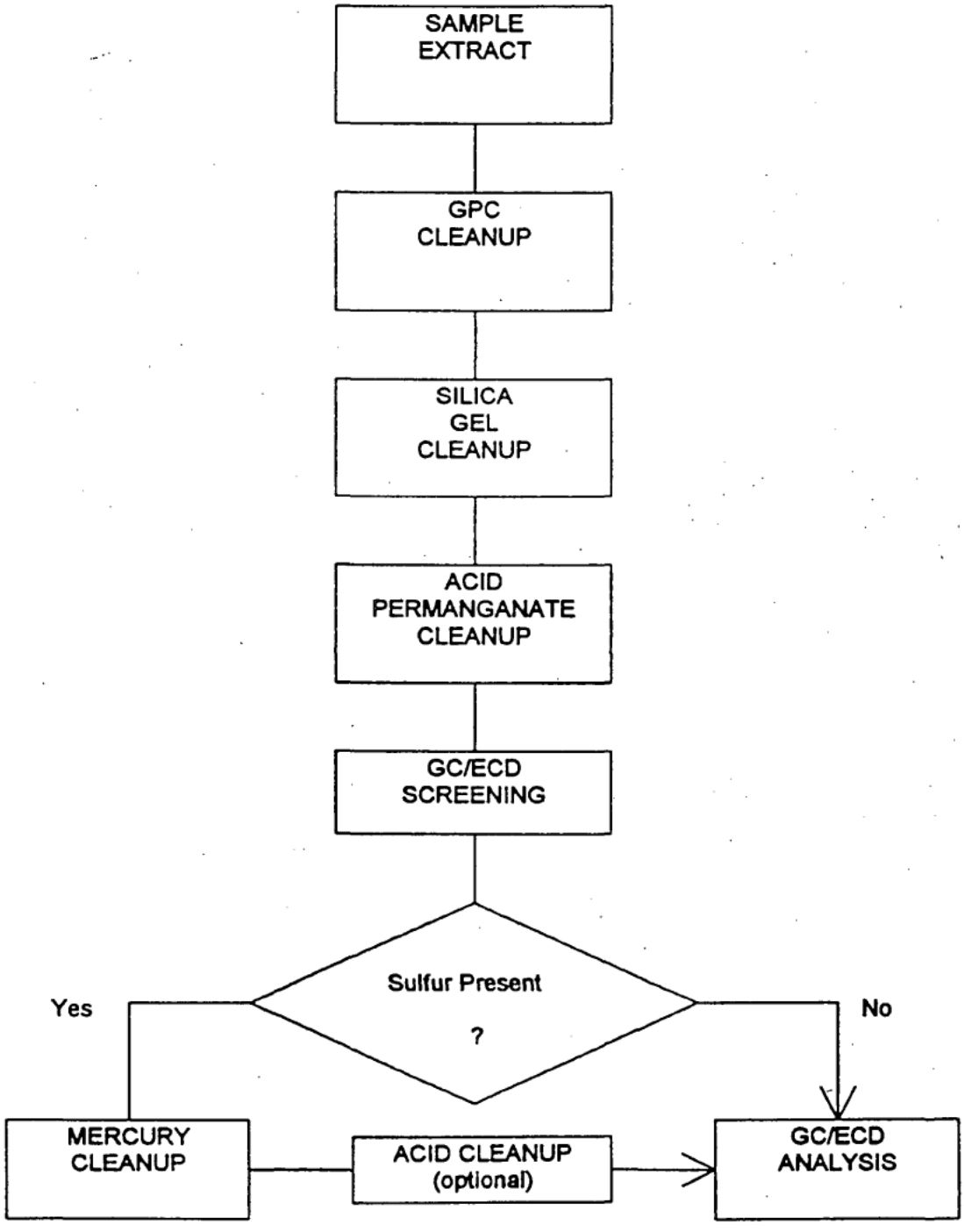

TABLE 1

Target Compound List

Compound Name	PCB I.D.	Water MRL (ng/L)	Water MDL (ng/L)	Soil/Sed MRL (ug/kg)	Soil/Sed MDL (ug/kg)
2,4'-Dichlorobiphenyl	PCB 8	5	0.8	0.5	0.4
2,2',5-Trichlorobiphenyl	PCB 18	5	0.7	0.5	0.2
2,4,4'-Trichlorobiphenyl	PCB 28	5	0.6	0.5	0.09
2,2',5,5'-Tetrachlorobiphenyl	PCB 52	5	0.3	0.5	0.07
2,2',3,5'-Tetrachlorobiphenyl	PCB 44	5	0.3	0.5	0.07
2,3',4,4'-Tetrachlorobiphenyl	PCB 66	5	0.7	0.5	0.09
2,3,4,4'-Tetrachlorobiphenyl	PCB 60	5	-	0.5	0.3
2,2',3,4',5-Pentachlorobiphenyl	PCB 90	5	0.8	0.5	0.2
2,2',4,5,5'-Pentachlorobiphenyl	PCB 101	5	0.4	0.5	0.2
3,4,4',5-Tetrachlorobiphenyl	PCB 81	5	-	0.5	0.06
2,2',3,4,5'-Pentachlorobiphenyl	PCB 87	5	0.2	0.5	0.07
3,3',4,4'-Tetrachlorobiphenyl	PCB 77	5	0.3	0.5	0.3
2',3,4,4',5-Pentachlorobiphenyl	PCB 123	5	0.2	0.5	0.07
2,3',4,4',5-Pentachlorobiphenyl	PCB 118	5	0.2	0.5	0.07
2,3,4,4',5-Pentachlorobiphenyl	PCB 114	5	0.3	0.5	0.1
2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB 184	5	0.2	0.5	0.08
2,2',4,4',5,5'-Hexachlorobiphenyl	PCB 153	5	0.2	0.5	0.2
2,3,3',4,4'-Pentachlorobiphenyl	PCB 105	5	0.2	0.5	0.4
2,2',3,4,4',5'-Hexachlorobiphenyl	PCB 138	5	0.2	0.5	0.3
2,3,3',4,4',6-Hexachlorobiphenyl	PCB 158	5	-	0.5	0.07
3,3',4,4',5-Pentachlorobiphenyl	PCB 126	5	0.2	0.5	0.2
2,3,4,4',5,6-Hexachlorobiphenyl	PCB 166	5	-	0.5	0.1
2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB 187	5	0.2	0.5	0.2
2,2',3,4,4',5',6-Heptachlorobiphenyl	PCB 183	5	0.4	0.5	0.08
2,2',3,3',4,4'-Hexachlorobiphenyl	PCB 128	5	0.3	0.5	0.2
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB 167	5	0.4	0.5	0.2
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 156	5	0.4	0.5	0.09
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 157	5	0.2	0.5	0.07
2,2',3,4,4',5,5'-Heptachlorobiphenyl	PCB 180	5	0.2	0.5	0.3
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB 169	5	0.3	0.5	0.09
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB 170	5	0.3	0.5	0.3
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB 189	5	0.3	0.5	0.08
2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB 195	5	0.3	0.5	0.07
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB 206	5	0.5	0.5	0.07
Decachlorobiphenyl	PCB 209	5	0.4	0.5	0.08

TABLE 2

Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Hewlett-Packard Model 5890 or equivalent w/ECD
Injection Port Temperature:	Cool on-column, oven track
Oven Temperature Program:	60°C for one minute, 40°/min ramp to 190°C, hold 25 min., ramp 1.2°/minute to 210° C, hold for 25 minutes, ramp 5.0°/minute to 280° C, hold for 10 minutes.
Detector Temperature:	350°C
Injection Volume:	1µL
Column:	30 m, RTX-5 and 30 m RTX-1701.
Carrier Gas:	Helium, programmed: 99psi/min. to 40psi, hold .2 min., then 99psi/min to 19psi (constant pressure).
Auxillary Gas:	Argon/Methane
Data System:	HP Enviroquant

* The instrument temperatures may be modified depending on the instrument used. Also, the GC column diameter and film thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analyses.

APPENDIX I

QC Acceptance Limits

SURROGATE SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
2,2',4,4',6,6'-Hexabromobiphenyl	30-150	30-150

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Compound	Water	Soil/Sediment
PCB 8	30-150	30-150
PCB 18	30-150	30-150
PCB 44	30-150	30-150
PCB 123	30-150	30-150
PCB 128	30-150	30-150
PCB 170	30-150	30-150
PCB 195	30-150	30-150
PCB 206	30-150	30-150
PCB 209	30-150	30-150

MATRIX SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Matrix Spike Compound	Water	Soil/Sediment
PCB 8	30-150	30-150
PCB 18	30-150	30-150
PCB 44	30-150	30-150
PCB 123	30-150	30-150
PCB 128	30-150	30-150
PCB 170	30-150	30-150
PCB 195	30-150	30-150
PCB 206	30-150	30-150
PCB 209	30-150	30-150

Attachment A

CAS Organics Calibration Policy

Columbia Analytical Services Inc.
An Employee-Owned Company

MEMORANDUM

DATE: May 19, 1999

TO: Department Managers, Kelso Organics

FROM: Joe Wiegel

SUBJ: **INITIAL AND CONTINUING CALIBRATIONS FOR ORGANICS, DRAFT REVISION NO. 9**

This memo states our policy on performing and evaluating initial and continuing calibrations for organic analyses. The guidelines stated in the memo are designed as default specification. Unless stated in standard operating procedures and prescribed in the determinative method, this memo states our standard operating procedure for calibrations. This policy is consistent with EPA Method 8000B, SW-846, Third Edition, Update III.

1. General Calibration Guidelines

   1.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

   1.2. Calibrations for organic analyses must contain a minimum of five concentrations.

   1.3. The method reporting limit (MRL) must be supported by the calibration, typically as the low point in the calibration.

   1.4. The complete calibration (i.e., all initial calibration (ICAL) levels and the independent calibration verification (ICV) standard) must be analyzed prior to analysis of field or QC samples.

   1.5. A calibration may not be interrupted by a maintenance event.

   1.6. A calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated.

   1.7. Calibration points may be dropped at the endpoints of the curve as long as conditions 1.1, 1.2 and 1.3 are met.

1.8. Calibration points may not be dropped from the interior of a curve unless a catastrophic error (e.g., gross dilution error, missing internal standards, injection malfunction, etc.) is accounted for in a nonconformity and corrective action report (NCAR). In these circumstances, all the analytes in that calibration standard must be dropped from the calibration curve as corrective action.

1.9. Analysis of all field and QC samples must be preceded by an acceptable ICAL and ICV, or must be preceded by an acceptable CCV that verifies the ICAL.

2. Evaluation Guidelines for Initial Calibrations

2.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

2.2. Average response factor (RF*ave*) is the preferred calibration technique because linearity through the origin is assumed. As such, this technique allows the analyst to perform a more intuitive assessment of data below the lowest calibration standard. However, RF*ave* may not always be the best fit of calibration data. The analyst should use prior knowledge of the instrument, analyte response, and an assessment of the calibration data in determining whether RF*ave* is appropriate.

2.2.1. Acceptance criteria for RF*ave*:

GC and HPLC

2.2.1.1. Relative standard deviation (RSD) equal to or less than 20% for all compounds.

GC/MS

2.2.1.2. System performance and calibration check compounds (SPCC and CCC) must meet method criteria.

2.2.1.3. Relative standard deviation (RSD) equal to or less than 15% for all compounds.

Allowable Exceptions

2.2.1.4. Some analytes are recognized as marginal performing compounds. Typically, these are analytes that are not expected to meet the primary evaluation criteria due to the chemical nature of the compound. Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. The list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be indicated in the analytical SOP. Acceptance criteria for RF*ave* for these compounds are as follow:

GC and HPLC Procedures

2.2.1.4.1. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%

2.2.1.4.2. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 20%.

2.2.1.4.3. The RSD for marginal performing compounds must be less than or equal to 40%.

GC/MS Procedures

2.2.1.4.4. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

2.2.1.4.5. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.1.4.6. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 15%.

2.2.1.4.7. The RSD for marginal performing compounds must be less than or equal to 30%.

2.2.2. A calibration that has been processed using RF*ave* that does not meet the criteria described above may be used to report non-detected analytes. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. The following criteria apply:

2.2.2.1. For GC and HPLC procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%.

2.2.2.2. For GC/MS procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.2.3. Adequate sensitivity to detect and confirm the analyte at the MRL must be demonstrated by the lowest calibration standard.

2.2.2.4. All confirmed detections for analytes exceeding RSD criteria in the ICAL, regardless of concentration, must be reanalyzed to ensure accurate quantification following recalibration of the instrument.

2.3. When curves are used, least squares and quadratic fits are permissible. These fits will not be forced through the origin. As such, it may become impractical to report estimated concentrations in samples below the lowest standard of calibration. When used, the analyst must be trained on the significance of the Y-intercept when using curve fits. Specifically, the analyst must understand that very low responses may

quantify to false positives depending on where the curve intersects the Y axis. Acceptance criteria for these fits are as follow:

2.3.1. Least Squares: $R \geq 0.995$ or $R^2 \geq 0.990$

2.3.2. Quadratic: $COD \geq 0.990$

2.3.3. If a least squares fit is used, the curve must contain a minimum of five (5) calibration levels.

2.3.4. If a quadratic fit is used, the curve must contain a minimum of six (6) calibration levels and must be continuous along the function (i.e., it must consist of consecutive increasing or consecutive decreasing numerical values).

2.4. Because calibration curves can not be forced through the origin, the analyst should evaluate the effect the y-intercept will have on quantifying detections at or below the lowest standard of calibration. This evaluation should involve extrapolating the curve to a level of one half the lowest standard (using one half the area or area ratio). If the result is positive and less than the MRL, the curve may be used. If the result is equal to or less than zero, or if the result is equal to or greater than the MRL, the curve fit is not to be used.

2.5. The calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated. Acceptance criteria for all techniques (GC, HPLC and GC/MS) are as follow:

2.5.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

2.5.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

2.5.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±30% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

2.5.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. ICV acceptance criteria on these analytes is ±30% for GC and HPLC procedures and ±40% for GC/MS procedures.

3. Evaluation Criteria: Continuing Calibration Verification (CCV) Standards

3.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

3.2. For calibrations using RF*ave*, CCV Standards are evaluated based on % Difference (%Dif) of the response factor. %Dif is calculated as:

$$\%Dif = \frac{RFv - RFave}{RFave} \times 100$$

Where:

RFv is the response factor (also relative response factor) or calibration factor from the verification standard and ***RFave*** is the average response factor or calibration factor from the calibration.

3.3. For calibrations using Least Squares or Quadratic fits, CCV Standards are evaluated based on % Drift (%Drft) of the measured value compared to the expected value. %Drft is calculated as:

$$\%Drft = \frac{Measured\ concentration - Expected\ concentration}{Expected\ concentration} \times 100$$

3.4. CCV Acceptance Criteria:

GC and HPLC Procedures

3.4.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±15% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

3.4.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±30%.

GC/MS Procedures

3.4.5. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

3.4.6. The average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.7. For individual analytes, the maximum allowable %Dif or %Drft is ±20% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.8. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±40%.

3.5. Non-detected analytes can be reported from analytical sequences that contained CCVs that do not pass acceptance criteria. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. This includes reanalysis of QA/QC samples containing spiked amounts of target analyte. The following criteria apply:

3.5.1. For GC and HPLC procedures, the average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.2. For GC/MS procedures, the average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.3. The analysis must demonstrate adequate sensitivity to detect and confirm the analyte at the MRL. Therefore, all analytes being reported to the client can not exhibit a %Dif or %Drft greater than ±40%. For external calibrations, this criteria applies to both preceding and concluding CCVs.

3.5.4. All confirmed detections for analytes that fail acceptance criteria in the CCV, regardless of concentration, must be reanalyzed to ensure accurate quantification. This criteria applies to all samples and QA/QC samples analyzed in the sequence.

3.6. CCV standards must be analyzed at the start of each analytical sequence (except when the sequence is initiated with an ICAL and ICV).

3.7. Two sequential analyses of a CCV at the beginning of an analytical sequence can be performed in an effort to prime the instrument for analysis. Routine analysis of two or

more sequential CCV standards throughout the analytical sequence in an effort to ensure continuation of the sequence is not permitted.

3.8. CCV standards are analyzed at the following frequency:

External Standard Calibrations

3.8.1. CCV standards should be analyzed after every 10 injections of field and QC samples or every 12 hours, which ever is more frequent.

3.8.2. Samples with confirmed detections must be bracketted by acceptable CCV standards.

3.8.3. When a closing CCV standard is not acceptable, corrective action must be taken. A new CCV standard may be prepared and analyzed to demonstrate degradation of the standard as the cause of a CCV outlier. In this case, instument stability will be verified and samples analyzed prior to this CCV can be reported. CCV standards that are reinjected after minor instrument maintenance (e.g., injection port maintenance, column bake-out, installation of a new trap, etc.) do not verify instrument stability. Samples analyzed prior to this CCV must be reanalyzed.

Internal Standard Calibrations

3.8.4. The analysis sequence must be initiatied with an acceptable instrument tune (for GC/MS) and an acceptable CCV. The last injection or analysis in the sequence must be started within 12 hours from the time of sequence initiation.

3.8.5. Internal standard response in the CCV should be within 0.5 - 2 times the response observed in the mid point calibration standard in the curve. Failure to meet this criteria should prompt the analyst to check the internal standard solution for possible degradation or concentration, or may indicate the need for instrument maintenance.

3.8.6. Internal standard response in each sample must be within 0.5 - 2 times the response observed in the initial CCV standard of the analytical sequence (or the mid point calibration standard in the curve if the analytical sequence began with an ICAL). Perform reanalysis or dilutions to assess the effect of matrix interferences on internal standard responses.

STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED-MASS SPECTROMETRY (ICP-MS)

SOP No.: MET-ICPMS

Revision: 1

May 24, 1999

Approved by: _____ Supervisor — 5-25-99 Date

_____ QA Manager — 5-25-99 Date

_____ Laboratory Manager — 5-25-99 Date

1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

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DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED-MASS SPECTROMETRY (ICP-MS) - METHOD 200.8

1. SCOPE AND APPLICATION

1.1. This procedure describes the steps taken for the analysis of soil, sludge and water digestates using EPA ICP method 200.8 for a variety of elements. This SOP is intended to be used in conjunction with the EPA method as a guide to ICP-MS analysis. The complexity of the technique generally requires outside study of appropriate literature as well as specialized training by a qualified spectroscopist. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

1.2. The Method Reporting Limits (MRLs) for common elements are listed in Table 1. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. Method Detection Limits (MDLs) which have been achieved are listed in Table 1.

2. METHOD SUMMARY

2.1. Discussion:

2.1.1. Prior to analysis, samples must be digested using appropriate sample preparation methods. The digestate is analyzed for the elements of interest using ICP spectrometry.

2.1.2. Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2.2. Deviations from the reference method(s): This SOP contains no deviations from the reference methods.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2. **Independent Calibration Verification (ICV)** - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3. **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.4. **Duplicate Sample** (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.5. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.6. **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.7. **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

4. INTERFERENCES

4.1. Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isoptope.

4.2. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature.

5. SAFETY

5.1. Corrosives - Because all samples and standards are generally diluted in 1% HNO3, there is a danger of exposure to corrosives. Sufficient care must be taken in handling the solutions. Safety glasses must be worn while preparing and handling the solutions.

5.2. High Voltage - The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

5.3. UV Light - The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Samples are generally received in the ICP-MS laboratory as 1% Nitric Acid digestates. Samples are stored in the appropriate volumetric containers.

6.2. Digestates originating from soil samples with greater than 60% solids are diluted prior to instrumental analysis by a factor of 5. This allows the analysis to achieve maximum sensitivity which results in optimum Method Reporting Limits (MRL). Digestates are diluted and disposed of through the sewer system 2 weeks after data is reviewed. Following analysis, digestates are stored until all results have been reviewed.

7. APPARATUS & EQUIPMENT

Instrument:	VG PlasmaQuad II Turbo-Plus
Nebulizer:	TJA Fixed Crossflow
Spray Chamber:	VG Water-cooled
Cones:	Nickel Sampler (1.0 mm orifice)
	Nickel Skimmer (0.75 mm orifice)
Peristaltic Pump	

8. STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

8.1. All standards are prepared from NIST traceable standards as per SOP Std. Prep. Manufacturers expiration dates are used to determine viability of standards.

8.1.1. Standard Log

8.1.1.1. The manufacturer, lot number, and expiration date are recorded on a standard log (see Appendix I). A copy of this is kept in the ICP-MS lab. For all Tier III reports, a copy of the standard information is included with the data package.

8.1.1.2. The lot number and date opened for acids used are recorded on the standard log.

8.1.1.3. The operator who prepares the standard makes a copy of the form and places it in the standards log book. The operator also places his initials and the date prepared on the standard container.

8.1.2. Working Standards

8.1.2.1. These are prepared from a pre-mixed stock solution. Documentation for these standards is the same as in Section 8.1.

8.1.3. Continuing Calibration Verification (CCV) Standard

The CCV standard is prepared daily from the working standards. The CCV is made up of equal parts of the standards. If more than two standards (not including the blank standard) are used, a separate CCV must be used for the third standard.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance is documented in the instrument log book. CAS/Kelso maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor.

9.2. Most routine maintenance and troubleshooting is performed by CAS staff, factory trained by the instrument manufacturer. Preventative maintenance activities listed below are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. The instrument manual gives instructions for routine maintenance activities.

9.2.1. Daily Maintenance

- cone removal and cleaning

9.2.2. Weekly Maintenance

- removal and cleaning of the ICP glassware and fittings
- inspecting and cleaning the nebulizer

- checking the RF contact strip
- checking the water filter and replacing if necessary
- checking the air filters and cleaning if necessary

9.2.3. Monthly Maintenance

- Remove and clean the extraction lens
- check the rotary pump oil and add if necessary
- check the oil mist filters and clean if necessary

9.2.4. Semi-annual Maintenance

- clean the ion lens stack
- check the penning gauge and clean if necessary
- change the rotary pump oil

9.2.5. Annual Maintenance

- perform manual inspection of slide valve

9.2.6. Other Maintenance

- replace the electron multiplier tube when sensitivity degrades below acceptable levels

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the applicable EPA method, is also the responsibility of the department supervisor/manager.

11. PROCEDURES

11.1. Refer to method 200.8 (Appendix II) or the instrument manual for detailed instruction on implementation of the following daily procedures preceding an analytical run.

11.2. After the instrument has been placed in the "Operate" mode, begin completing the daily instrument log (App.III). The following parameters need to pass the listed acceptance criteria prior to continuing.

11.2.1. Pressures (Operate state)

Analyzer:	1-5 x E-10 mbar
Intermediate:	<0-1 x E-4 mbar
Expansion:	1-2 x E0 mbar

11.2.2. ICP Generator

Forward Power:	1350 watts
Plate Current:	0.68-0.72
Grid Current:	0.18-0.22
PA Voltage:	0.40-0.42
PA Filament:	0.79-0.81
Reflected Power:	<10

11.2.3. Water:

Water Temp:	10-12 C
Water Level:	Full

11.3. The following parameters are monitored to assure awareness of changes in the instrumentation that serve as signals that optimum performance is not being achieved, or as indicators of the physical condition of certain consumable components (i.e. EMT and cones).

11.3.1. Multiplier High Voltage

11.3.2. Record HT1 setting. As the EMT ages, the voltage applied will need to be increased to maintain sensitivity. See instrument manual for details.

11.3.3. Record HT2 setting. CAS does not utilize the low sensitive detection mode (analog) controlled by the HT2 setting. Proper setting of the voltage, however, is necessary due to the interaction of the two. When HT1 adjustment becomes necessary, the HT2 adjustment should also be performed at the same time.

11.3.4. Gas Flows

11.3.5. Coolant Ar: 14 L/min

11.3.6. The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.4. Optimization

11.4.1. Gas Flows

Aspirate a 25 ppb In solution into the plasma and monitor the instrument output signal at mass 114 on the ratemeter. Adjust the nebulizer and auxiliary flows to obtain maximum signal. Adjust the tension screw on the peristaltic pump to obtain minimum noise in the analytical signal. Record flow rates and note any large variances.

Note: Significant differences in flow rates will be observed for different torches and cones.

11.4.2. Ion Lens Setting

While monitoring the output signal of a 25 ppb In solution at mass 114 on the ratemeter, adjust the ion lenses to obtain maximum sensitivity. Refer to the instrument manual for details on performing the adjustments. The acceptable ranges for each lens are listed below. If operating outside these ranges, consult the troubleshooting section of the instrument manual. Expected values are listed in parenthesis.

Extraction:	0.00-2.50	(1.00)
Collector:	0.00-9.00	(7.70)
L1	6.50-9.00	(7.70)
L2	1.90-6.60	(5.40)
L3	0.00-7.75	(5.00)
L4	0.00-6.60	(3.80)
Pole bias	4.50-7.00	(6.00)

11.4.3. Mass Calibration

Aspirate a 25 ppb solution of Be, Mg, Co, In, La, Pb, and U using the Mass Calibration program in the VG software with these elements identified in the program as the points used for mass calibrating. Refer to the instrument manual for details pertaining to the mass calibration procedure.

11.4.4. Resolution Check

Using the spectra created during the mass calibration procedure, perform the resolution check as per EPA Method 200.8. Acceptance criteria are defined in the method.

11.4.5. Stability Check

Using the 25 ppb mixed element solution from the mass calibration check, perform a short-term stability check as per EPA Method 200.8. Acceptance criteria are defined in the method.

11.5. Analytical Run

11.5.1. Select the correct method.

11.5.2. Nebulize Standard 0 (Blank) into the plasma. Allow 1-2 minutes for system to equilibrate prior to establishing baseline.

11.5.3. Follow directions on computer screen to perform standardization. Operator will sign and date the first page of standardization.

11.5.4. Perform the analysis in the order listed below.

Initial Calibration Verification (ICV)
Continuing Calibration Verification (CCV)
Continuing Calibration Blank (CCB)
CRA - 1ppb or project specified level*
Analyze 9 Samples
CCV
CCB
Analyze 10 Samples
CCV
CCB
Repeat sequence as required to complete analytical run

* Note: For AFCEE projects, the MRL standard concentrations will be equal to the project MRLs.

12. QUALITY CONTROL

12.1. A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed after every 10 samples. The control limits are listed in EPA Method 200.8 are used.

12.2. The baseline or slope can be corrected provided the previous and following CCV and CCB are within acceptable limits. If CCV or CCB are not within acceptable limits, the analysis is terminated, the problem found, and the instrument is then restandardized.

12.3. As per method 200.8, a digested duplicate and matrix spike are analyzed at a frequency of 10% or two per batch of up to 20 samples, whichever is greater. The matrix spike

recovery and relative percent difference will be calculated while analysis is in progress. The control limits listed in the current EPA CLP SOW for Inorganics are used.

12.4. Laboratory Control Samples are analyzed at a frequency of 5% or one per batch, whichever is greater. The control limits listed in the current EPA CLP Statement of Work for Inorganics are used.

12.5. Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), and linear ranges are performed periodically as per Method 200.8. These will be calculated and made available to the ICP-MS operator.

12.6. Common isobaric interferences are corrected using equations equivalent to those listed in EPA Method 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. The scope of this document does not allow for an in-depth discussion/training course pertaining to the interpretation of ICP-MS spectra. The final review and approval of all data is performed by qualified spectroscopists. Refer to the Interferences section of EPA Method 200.8 for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

12.7. Refer to the Quality Control section of EPA Method 200.8 for additional information describing required QA/QC. Note that the nomenclature of certain QC samples in the method differs from that of the CLP SOW, but the function of those samples is equivalent in both cases.

13. DATA REDUCTION, REPORTING, AND REVIEW

13.1. Calculations

13.1.1. Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the caluculations below to generate a sample result.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = C^{*} \times Digestion\ Dilution\ Factor \times Post\ Digestion\ Dilution\ Factor$$

Solid samples are reported in mg/Kg:

$$mg / Kg\ (Sample) = C^{*} \times Post\ Digestion\ Dilution\ Factor \times \frac{Digestion\ Vol.\ (ml)}{Sample\ wt.\ (g)} \times \frac{1mg}{1000g} \times \frac{1L}{1000ml} \times \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L (in digestate).

Appendix I

App. I

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 5% HNO3 (Optima)
VOLUME: 1000 ml.
DATE: 2/24/94
ANALYST: Greg Jaeger

ELEMENT	SOURCE	LOT #	EXPIRATION DATE	ALIQUOT PER 1000 ml.	CHECK OFF	CONCENTRATION (µg/L)
HNO3	Fischer	7697-37-2	-	50.0 ml.	✓	5%
Al	SPEX	3-58AL	10/31/94	1.0 ml.	✓	1000
Sb	SPEX	2-216SB	8/15/94	1.0 ml.	✓	1000
As	I.V. *	I-AS0146	10/1/94	1.0 ml.	✓	1000
Ba	I.V.	I-BA0152	9/1/94	1.0 ml.	✓	1000
Be	I.V.	I-BE0133	10/1/94	1.0 ml.	✓	1000
Cd	SPEX	3-26CD	8/15/94	1.0 ml.	✓	1000
Cr	SPEX	3-22CR	8/30/94	1.0 ml.	✓	1000
Co	I.V.	H-CO0135	10/1/94	1.0 ml.	✓	1000
Cu	I.V.	I-CU0166	10/1/94	1.0 ml.	✓	1000
Pb	I.V.	H-PB0150	7/1/94	1.0 ml.	✓	1000
Mn	SPEX	2-26MN	8/15/94	1.0 ml.	✓	1000
Mo	I.V.	I-MO0139	11/1/94	1.0 ml.	✓	1000
Ni	SPEX	2-266NI	9/15/94	1.0 ml.	✓	1000
Se	I.V.	I-SE01030	12/1/94	1.0 ml.	✓	1000
Tl	I.V.	I-TL01035	2/1/94	1.0 ml.	✓	1000
U	I.V.	H-U0120	5/1/94	1.0 ml.	✓	1000
V	SPEX	2-233V	8/15/94	1.0 ml.	✓	1000
Zn	I.V.	I-ZN0151	9/1/94	1.0 ml.	✓	1000

* I.V. = Inorganic Ventures

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3 (Optima)
VOLUME: 1000 ml.
DATE: 8/26/93
ANALYST: Jeffrey A Carr

ELEMENT	SOURCE	LOT #	EXPIRATION DATE	ALIQUOT PER 250 ml.	CHECK OFF	CONCENTRATION (µg/L)
HNO3	Fischer	-	-	12.5	✓	5%
Ag	I.V.	H-AG0130	5/1/94	0.250	✓	1000

SOLUTION: ICP-MS, WORKING STANDARD #1
MATRIX: 1% HNO3 (Optima)
VOLUME: 1000 ml.
DATE:
ANALYST:

SOURCE	ALIQUOT PER 100 ml.	CHECK OFF	CONCENTRATION (µg/L)
HNO3 (Optima)	1.0		1%
IMTERMEDIATE STOCK	2.5		25.0
SILVER IMTERMEDIATE STOCK	2.5		25.0

Appendix II

TABLE 14: SPIKE MEASUREMENTS IN PARTICIPANT'S WASTEWATER*

	Concentrate 1					Concentrate 2					
	Spike	Found	Std Dev	%Rec	RSD	Spike	Found	Std Dev	%Rec	RSD	RSD,
	µg/L	µg/L	µg/L	%	%	µg/L	µg/L	µg/L	%	%	%
Be	101	103.4	12.0	103.4	11.6	125	128.2	13.6	102.6	10.6	2.4
Al	200	198.7	23.9	99.4	12.0	250	252.4	15.5	101.0	6.1	2.9
Cr	200	205.4	12.3	102.7	6.0	250	253.4	15.4	101.4	6.1	1.1
V	250	246.5	4.4	98.6	1.8	200	196.8	2.8	98.4	1.4	2.0
Mn	125	119.0	5.4	95.2	4.5	100	95.5	4.3	95.5	4.5	0.8
Co	125	125.8	7.0	100.6	5.6	101	99.5	5.3	98.5	5.3	1.8
Ni	125	127.4	9.7	101.9	7.6	100	101.0	7.5	101.0	7.4	1.7
Cu	125	126.8	5.3	101.4	4.2	100	105.3	3.6	105.3	3.4	2.8
Zn	200	201.4	36.7	100.7	18.2	250	246.4	29.7	98.6	12.1	2.6
As	200	207.3	11.9	103.7	5.7	250	263.0	2.6	105.2	1.0	3.2
Se	250	256.8	26.4	102.7	10.3	200	214.	18.7	107.3	8.7	3.6
Mo	100	98.6	4.6	98.6	4.7	125	123.2	6.7	98.6	5.4	2.2
Ag	200	200.7	48.9	100.4	24.4	250	231.2	63.5	92.5	27.5	8.2
Cd	125	123.2	11.5	98.6	9.3	100	95.8	2.9	95.8	3.0	5.8
Sb	100	92.2	4.4	92.2	4.8	125	119.0	1.0	95.2	0.8	2.8
Ba	250	245.2	12.8	98.1	5.2	200	204.7	12.1	102.4	5.9	2.1
Tl	100	100.0	0.9	100.0	0.9	125	128.0	6.0	102.4	4.7	3.5
Pb	125	125.8	5.1	100.6	4.1	100	100.8	2.7	100.8	2.7	2.2
Th	125	124.2	7.6	99.4	6.1	100	99.8	5.7	99.8	5.7	3.2
U	125	130.4	10.3	104.3	7.9	100	106.4	6.8	106.4	6.4	2.3

*Results from 5 participating laboratories. Mean concentrations before spiking are not listed because they varied considerably among the different wastewaters.

TABLE 12: SUMMARY STATISTICS AND DESCRIPTIVE EQUATIONS FOR THE 20 ANALYTES TESTED IN THE COLLABORATIVE STUDY

		Reagent Water				Finished Drinking Water				Ground Water			
Analyte	C[a]	$\bar{X}$[b]	S_R	S_r	Regr. Equations	$\bar{X}$	S_R	S_r	Regr. Equations	$\bar{X}$	S_R	S_r	Regr. Equations
Uranium	0.80	0.86	0.05	0.08	$\bar{X} = 1.026\underline{C} - 0.02$	0.85	0.15	0.09	$\bar{X} = 1.026\underline{C} - 0.04$	0.84	0.23	0.19	$\bar{X} = 1.058\underline{C} - 0.06$
	1.20	1.10	0.11		$S_R = 0.048\underline{X} + 0.02$	1.05	0.13		$S_R = 0.044\underline{X} + 0.11$	1.10	0.14		$S_R = 0.039\underline{X} + 0.17$
	20.10	21.38	0.99	0.82	$S_r = 0.027\bar{X} + 0.05$	22.30	1.40	0.46	$S_r = 0.022\bar{X} + 0.07$	21.56	1.11	1.08	$S_r = 0.028\bar{X} + 0.16$
	28.10	28.36	1.10			28.89	1.47			29.86	1.83		
	80.30	82.47	4.03	2.16		80.31	2.00	2.71		85.01	3.76	2.00	
	100.00	103.49	5.24			100.70	5.30			106.47	3.74		
Vanadium	32.00	31.02	2.68	2.19	$\bar{X} = 1.025C - 2.21$	33.15	2.51	2.28	$\bar{X} = 1.022\underline{C} - 0.30$	33.25	3.83	1.87	$\bar{X} = 1.076\underline{C} - 1.87$
	40.00	38.54	2.94		$S_R = 3.79$[d]	40.20	1.88		$S_R = 0.023\underline{X} + 1.45$	40.34	3.08		$S_R = 0.033\underline{X} + 2.25$
	80.00	79.14	4.94	4.29	$S_r = 3.26$[d]	77.83	4.18	2.75	$S_r = 0.023\bar{X} + 1.38$	84.42	3.97	2.93	$S_r = 0.049\bar{X} - 0.09$
	96.00	93.47	3.85			96.32	1.34			98.70	5.03		
	160.00	162.43	5.67	3.30		161.89	7.63	6.56		170.94	9.09	11.55	
	200.00	208.20	2.65			214.91	5.89			217.90	11.36		
Zinc	8.00	8.33	2.56	1.78	$\bar{X} = 1.042\underline{C} + 0.87$	11.60	6.18	5.72	$\bar{X} = 0.943\underline{C} + 2.54$	7.29	1.12	2.20	$\bar{X} = 0.962\underline{C} + 0.07$
	12.00	15.49	4.18		$S_R = 0.041\underline{X} + 2.60$	10.21	4.96		$S_R = 0.048\underline{X} + 5.27$	12.66	3.24		$S_R = 0.093\underline{X} + 0.92$
	56.00	56.07	2.91	2.47	$S_r = 0.030\bar{X} + 1.42$	56.83	7.66	4.56	$S_r = 0.004\bar{X} + 5.66$[e]	54.86	5.12	7.24	$S_r = 0.069\bar{X} + 1.55$
	80.00	85.53	5.81			82.88	8.34			78.62	8.56		
	160.00	165.17	7.78	9.87		156.69	17.01	9.48		150.12	12.52	10.84	
	200.00	207.27	14.61			191.59	17.21			184.37	16.59		

[a] True Value for the concentration added (µg/L)
[b] Mean Recovery (µg/L)
[c] $COD_r < 0.5$ - Use of regression equation outside study concentration range not recommended.
[d] $COD_r < 0$ - Mean precision is reported.
[e] $COD_r < 0$ - Unweighted linear regression equation presented.

TABLE 12: SUMMARY STATISTICS AND DESCRIPTIVE EQUATIONS FOR THE 20 ANALYTES TESTED IN THE COLLABORATIVE STUDY

		Reagent Water				Finished Drinking Water				Ground Water			
Analyte	C[a]	$\bar{X}$[b]	S_R	S_r	Regr. Equations	$\bar{X}$	S_R	S_r	Regr. Equations	$\bar{X}$	S_R	S_r	Regr. Equations
Chromium	8.00	8.27	0.32	1.54	$\bar{X} = 1.017C + 0.62$	9.46	2.34	2.08	$\bar{X} = 0.990C + 1.45$	8.98	1.47	0.37	$\bar{X} = 1.026C + 0.89$
	12.00	13.88	3.10		$S_R = 0.066\bar{X} + 0.48$	13.10	2.39		$S_R = 0.015\bar{X} + 2.19$	13.42	1.13		$S_R = 0.067\bar{X}$ 0.68
	56.00	57.86	4.03	2.68	$S_r = 0.026\bar{X} + 1.25$	56.04	2.24	1.29	$S_r = 2.18$[d]	59.35	5.99	5.42	$S_r = 0.068\bar{X} - 0.37$
	80.00	84.73	2.65			84.38	3.18			83.90	5.70		
	160.00	157.66	13.62	6.97		158.24	5.12	3.16		164.58	14.11	9.80	
	200.00	197.43	9.47			196.72	7.47			199.88	11.19		
Cobalt	0.80	0.88	0.10	0.05	$\bar{X} = 0.977C + 0.01$	0.92	0.45	0.31	$\bar{X} = 0.964C + 0.06$	0.85	0.13	0.09	$\bar{X} = 0.989C - 0.01$
	1.21	0.98	0.04		$S_R = 0.028\bar{X} + 0.06$	1.02	0.10		$S_R = 0.019\bar{X} + 0.32$	1.04	0.18		$S_R = 0.057\bar{X} + 0.09$
	20.10	20.77	0.74	0.67	$S_r = 0.027\bar{X} + 0.02$	20.45	0.91	0.53	$S_r = 0.014\bar{X} + 0.30$	20.81	1.11	1.12	$S_r = 0.012\bar{X} + 0.40$[e]
	28.20	27.75	0.96			27.29	1.22			28.07	2.16		
	80.50	78.59	2.29	2.31		78.04	3.72	1.84		79.26	4.66	1.34	
	101.00	98.79	2.94			97.62	4.62			99.41	4.22		
Copper	4.00	3.88	0.73	0.59	$\bar{X} = 1.003C - 0.05$	3.33	0.85	0.99	$\bar{X} = 0.976C - 0.38$	3.86	1.40	0.71	$\bar{X} = 0.977C - 0.01$
	6.00	6.14	1.00		$S_R = 0.037\bar{X} + 0.64$	5.95	1.78		$S_R = 0.063\bar{X} + 0.86$	5.96	0.95		$S_R = 0.073\bar{X} + 0.92$
	20.00	20.07	1.08	0.92	$S_r = 0.016\bar{X} + 0.51$	18.90	1.64	1.51	$S_r = 0.029\bar{X} + 0.86$	18.97	1.68	2.32	$S_r = 0.077\bar{X} + 0.35$
	28.00	27.97	1.94			27.21	2.76			27.44	2.58		
	80.00	79.80	3.22	1.91		76.64	5.30	3.42		79.30	9.05	6.54	
	100.00	99.57	4.42			96.17	5.64			97.54	11.16		
Lead	4.00	4.00	1.57	1.62	$\bar{X} = 1.043C - 0.31$	3.44	1.15	1.18	$\bar{X} = 1.032C - 0.30$	4.20	1.13	1.76	$\bar{X} = 1.012C + 0.15$
	6.00	5.56	2.00		$S_R = 0.064\bar{X} + 1.43$[e]	6.84	1.10		$S_R = 0.015\bar{X} + 1.06$	6.27	2.38		$S_R = 0.048\bar{X} + 1.27$
	20.00	20.54	2.91	4.36	$S_r = 3.42$[d]	20.18	1.20	1.44	$S_r = 0.011\bar{X} + 1.13$	19.57	2.72	0.88	$S_r = 1.78$[d]
	28.00	30.90	4.58			28.08	1.57			28.55	1.73		
	80.00	80.57	3.13	4.29		80.92	2.30	2.07		82.47	4.38	2.69	
	100.00	102.93	6.62			101.60	3.23			102.47	3.58		
Manganese	0.80	0.86	0.15	0.09	$\bar{X} = 0.983C + 0.02$	0.96	0.32	0.42	$\bar{X} = 0.989C + 0.10$	0.64	0.22	0.17	$\bar{X} = 0.954C - 0.16$
	1.20	1.09	0.12		$S_R = 0.026\bar{X} + 0.11$	1.13	0.38		$S_R = 0.047\bar{X} + 0.29$	0.90	0.21		$S_R = 0.103\bar{X} + 0.14$
	20.00	20.43	0.89	0.72	$S_r = 0.027\bar{X} + 0.06$	21.06	1.32	0.96	$S_r = 0.021\bar{X} + 0.40$	19.61	2.60	2.62	$S_r = 0.025\bar{X} + 0.09$[e]
	28.00	27.53	0.41			27.60	1.47			25.65	4.10		
	80.00	79.00	3.16	2.38		79.57	4.18	2.01		77.38	6.13	2.90	
	100.00	97.60	2.51			97.97	4.10			95.86	6.74		

TABLE 11 : PRIMARY DRINKING WATER CONTAMINANTS
PRECISION AND RECOVERY DATA

ANALYTE	REGIONAL SAMPLE BACKGROUND CONCENTRATION, µg/L (IV)	(VI)	(X)	AVERAGE MEAN[1] % RECOVERY	S(R)
Antimony	0.16	0.07	0.03	114%	1.9
Arsenic	< MDL	2.4	1.0	93	8.5
Barium	4.6	280	14.3	(*)	-
Beryllium	< MDL	< MDL	< MDL	100%	8.2
Cadmium	0.05	0.05	0.03	81	4.0
Chromium	0.71	5.1	0.10	94	2.5
Copper	208	130	14.3	(*)	-
Lead	1.2	1.2	2.5	91	2.6
Mercury	< MDL	0.23	< MDL	86	11.4
Nickel	1.7	3.6	0.52	101%	11.5
Selenium	< MDL	4.3	< MDL	98	8.4
Thallium	< MDL	0.01	< MDL	100	1.4

1 The three regional waters were fortified with 1.0 µg/L of all analytes listed, except selenium, which was fortified to 20 µg/L.

(*) Recovery of barium and copper was not calculated because the analyte addition was < 20% the sample background concentration in all waters. (Recovery calculations are not required if the concentration of the analyte added is less than 30% of the sample background concentration. Sect.9.4.3)

S(R) Standard deviation of the mean percent recoveries.

TABLE 10 : PRECISION AND RECOVERY DATA IN SOLID MATRICES (Cont).

NBS 1645 RIVER SEDIMENT

Element	Sample Concn. (mg/kg)	Low+ Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD	High+ Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD
Al	5060	20	*	*	-	100	*	*	-
Sb	21.8	20	73.9	6.5	9.3	100	81.2	1.5	3.9
As	67.2	20	104.3	13.0	7.6	100	107.3	2.1	2.9
Ba	54.4	20	105.6	4.9	2.8	100	98.6	2.2	3.9
Be	0.59	20	88.8	0.2	0.5	100	87.9	0.1	0.2
Cd	8.3	20	92.9	0.4	0.0	100	95.7	1.4	3.9
Cr	29100	20	*	*	-	100	*	*	-
Co	7.9	20	97.6	1.3	2.6	100	103.1	0.0	0.0
Cu	112	20	121.0	9.1	1.5	100	105.2	2.2	1.8
Pb	742	20	*	*	-	100	-	-	-
Mn	717	20	*	*	-	100	-	-	-
Mo	17.1	20	89.8	8.1	12.0	100	98.4	0.7	0.9
Ni	41.8	20	103.7	6.5	4.8	100	102.2	0.8	0.0
Se	<3.2	20	108.3	14.3	37.4	100	93.9	5.0	15.1
Ag	1.8	20	94.8	1.6	4.3	100	96.2	0.7	1.9
Tl	1.2	20	91.2	1.3	3.6	100	94.4	0.4	1.3
Th	0.90	20	91.3	0.9	2.6	100	92.3	0.9	2.8
U	0.79	20	95.6	1.8	5.0	100	98.5	1.2	3.5
V	21.8	20	91.8	4.6	5.7	100	100.7	0.6	0.8
Zn	1780	20	*	*	-	100	*	*	-

S(R) Standard deviation of percent recovery.
RPD Relative percent difference between duplicate spike determinations.
< Sample concentration below established method detection limit.
* Spike concentration <10% of sample background concentration.
- Not determined.
+ Equivalent.

TABLE 9 : PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES (Cont).

INDUSTRIAL EFFLUENT

Element	Sample Concn. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	44.7	50	98.8	8.7	5.7	200	90.4	2.1	2.2
Sb	2990	10	*	*	0.3	100	*	*	0.0
As	<1.4	50	75.1	1.8	6.7	200	75.0	0.0	0.0
Ba	100	50	96.7	5.5	3.4	200	102.9	1.1	0.7
Be	<0.3	10	103.5	1.8	4.8	100	100.0	0.0	0.0
Cd	10.1	10	106.5	4.4	2.4	100	97.4	1.1	2.8
Cr	171	10	*	*	0.0	100	127.7	2.4	1.7
Co	1.3	10	90.5	3.2	8.7	100	90.5	0.4	1.3
Cu	101	10	*	*	0.9	100	92.5	2.0	1.6
Pb	294	10	*	*	2.6	100	108.4	2.1	0.0
Mn	154	10	*	*	2.8	100	103.6	3.7	1.6
Mo	1370	10	*	*	1.4	100	*	*	0.7
Ni	17.3	10	107.4	7.4	5.0	100	88.2	0.7	1.0
Se	15.0	50	129.5	9.3	15.1	200	118.3	1.9	3.6
Ag	<0.1	50	91.8	0.6	1.7	200	87.0	4.9	16.1
Tl	<0.3	10	90.5	1.8	5.5	100	98.3	1.0	2.8
Th	0.29	10	109.6	1.2	2.7	100	108.7	0.0	0.0
U	0.17	10	104.8	2.5	6.6	100	109.3	0.4	0.9
V	<2.5	50	74.9	0.1	0.3	200	72.0	0.0	0.0
Zn	43.4	50	85.0	4.0	0.6	200	97.6	1.0	0.4

S(R) Standard deviation of percent recovery.
RPD Relative percent difference between duplicate spike determinations.
< Sample concentration below established method detection limit.
* Spike concentration <10% of sample background concentration.

TABLE 9 : PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES (Cont).

POND WATER

Element	Sample Concn. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	610	50	*	*	1.7	200	78.2	9.2	5.5
Sb	<0.4	10	101.1	1.1	2.9	100	101.5	3.0	8.4
As	<1.4	50	100.8	2.0	5.6	200	96.8	0.9	2.6
Ba	28.7	50	102.1	1.8	2.4	200	102.9	3.7	9.0
Be	<0.3	10	109.1	0.4	0.9	100	114.4	3.9	9.6
Cd	<0.5	10	106.6	3.2	8.3	100	105.8	2.8	7.6
Cr	2.0	10	107.0	1.0	1.6	100	100.0	1.4	3.9
Co	0.79	10	101.6	1.1	2.7	100	101.7	1.8	4.9
Cu	5.4	10	107.5	1.4	1.9	100	98.1	2.5	6.8
Pb	1.9	10	108.4	1.5	3.2	100	106.1	0.0	0.0
Mn	617	10	*	*	1.1	100	139.0	11.1	4.0
Mo	0.98	10	104.2	1.4	3.5	100	104.0	2.1	5.7
Ni	2.5	10	102.0	2.3	4.7	100	102.5	2.1	5.7
Se	<7.9	50	102.7	5.6	15.4	200	105.5	1.4	3.8
Ag	0.12	50	102.5	0.8	2.1	200	105.2	2.7	7.1
Tl	<0.3	10	108.5	3.2	8.3	100	105.0	2.8	7.6
Th	0.19	10	93.1	3.5	10.5	100	93.9	1.6	4.8
U	0.30	10	107.0	2.8	7.3	100	107.2	1.8	4.7
V	3.5	50	96.1	5.2	14.2	200	101.5	0.2	0.5
Zn	6.8	50	99.8	1.7	3.7	200	100.1	2.8	7.7

S(R) Standard deviation of percent recovery.
RPD Relative percent difference between duplicate spike determinations.
< Sample concentration below established method detection limit.
* Spike concentration <10% of sample background concentration.

TABLE 9 : PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES

DRINKING WATER

Element	Sample Concn. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	175	50	115.8	5.9	0.4	200	102.7	1.6	1.1
Sb	<0.4	10	99.1	0.7	2.0	100	100.8	0.7	2.0
As	<1.4	50	99.7	0.8	2.2	200	102.5	1.1	2.9
Ba	43.8	50	94.8	3.9	5.8	200	95.6	0.8	1.7
Be	<0.3	10	113.5	0.4	0.9	100	111.0	0.7	1.8
Cd	<0.5	10	97.0	2.8	8.3	100	101.5	0.4	1.0
Cr	<0.9	10	111.0	3.5	9.0	100	99.5	0.1	0.2
Co	0.11	10	94.4	0.4	1.1	100	93.6	0.5	1.4
Cu	3.6	10	101.8	8.8	17.4	100	91.6	0.3	0.3
Pb	0.87	10	97.8	2.0	2.8	100	99.0	0.8	2.2
Mn	0.96	10	96.9	1.8	4.7	100	95.8	0.6	1.8
Mo	1.9	10	99.4	1.6	3.4	100	98.6	0.4	1.0
Ni	1.9	10	100.2	5.7	13.5	100	95.2	0.5	1.3
Se	<7.9	50	99.0	1.8	5.3	200	93.5	3.5	10.7
Ag	<0.1	50	100.7	1.5	4.2	200	99.0	0.4	1.0
Tl	<0.3	10	97.5	0.4	1.0	100	98.5	1.7	4.9
Th	<0.1	10	109.0	0.7	1.8	100	106.0	1.4	3.8
U	0.23	10	110.7	1.4	3.5	100	107.8	0.7	1.9
V	<2.5	50	101.4	0.1	0.4	200	97.5	0.7	2.1
Zn	5.2	50	103.4	3.3	7.7	200	96.4	0.5	1.0

S(R) Standard deviation of percent recovery.
RPD Relative percent difference between duplicate spike determinations.
< Sample concentration below established method detection limit.

TABLE 7: METHOD DETECTION LIMITS

AMU ELEMENT	SCANNING MODE[1] TOTAL RECOVERABLE AQUEOUS µg/L	SCANNING MODE[1] SOLIDS mg/kg	SELECTIVE ION MONITORING MODE[2] TOTAL RECOVERABLE AQUEOUS µg/L	DIRECT ANALYSIS[3] AQUEOUS µg/L
^{27}Al	1.0	0.4	1.7	0.04
^{123}Sb	0.4	0.2	0.04	0.02
^{75}As	1.4	0.6	0.4	0.1
^{137}Ba	0.8	0.4	0.04	0.04
^{9}Be	0.3	0.1	0.02	0.03
^{111}Cd	0.5	0.2	0.03	0.03
^{52}Cr	0.9	0.4	0.08	0.08
^{59}Co	0.09	0.04	0.004	0.003
^{63}Cu	0.5	0.2	0.02	0.01
^{206,207,208}Pb	0.6	0.3	0.05	0.02
^{55}Mn	0.1	0.05	0.02	0.04
^{202}Hg	n.a.	n.a.	n.a.	0.2
^{98}Mo	0.3	0.1	0.01	0.01
^{60}Ni	0.5	0.2	0.06	0.03
^{82}Se	7.9	3.2	2.1	0.5
^{107}Ag	0.1	0.05	0.005	0.005
^{205}Tl	0.3	0.1	0.02	0.01
^{232}Th	0.1	0.05	0.02	0.01
^{238}U	0.1	0.05	0.01	0.01
^{51}V	2.5	1.0	0.9	0.05
^{66}Zn	1.8	0.7	0.1	0.2

1 Data acquisition mode given in Table 6. Total recoverable MDL concentrations are computed for original matrix with allowance for sample dilution during preparation. Listed MDLs for solids calculated from determined aqueous MDLs.

2 MDLs determined using state-of-the-art instrumentation (1994). Data for ^{75}As, ^{77}Se, and ^{82}Se were acquired using a dwell time of 4.096 sec with 1500 area count per sec ^{83}Kr present in argon supply. All other data were acquired using a dwell time of 1.024 sec per AMU monitored.

3 MDLs were determined from analysis of 7 undigested aqueous sample aliquots.

n.a.- not applicable. Total recoverable digestion not suitable for organo-mercury compounds.

TABLE 5 (Continued)

INTERNAL STANDARDS

Element	Elemental Equation	Note
Bi	$(1.000)(^{209}C)$	
In	$(1.000)(^{115}C)-(0.016)(^{118}C)$	(8)
Sc	$(1.000)(^{45}C)$	
Tb	$(1.000)(^{159}C)$	
Y	$(1.000)(^{89}C)$	

C - calibration blank subtracted counts at specified mass.
(1) - correction for chloride interference with adjustment for ^{77}Se. ArCl 75/77 ratio may be determined from the reagent blank. Isobaric mass 82 must be from Se only and not BrH^+.
(2) - correction for MoO interference. Isobaric mass 106 must be from Cd only not ZrO^+. An additional isobaric elemental correction should be made if palladium is present.
(3) - in 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank. Isobaric mass must be from Cr only not ArC^+.
(4) - allowance for isotopic variability of lead isotopes.
(5) - isobaric elemental correction for ruthenium.
(6) - some argon supplies contain krypton as an impurity. Selenium is corrected for ^{82}Kr by background subtraction.
(7) - correction for chloride interference with adjustment for ^{53}Cr. ClO 51/53 ratio may be determined from the reagent blank. Isobaric mass 52 must be from Cr only not ArC^+.
(8) - isobaric elemental correction for tin.

TABLE 4: RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MUST BE MONITORED

Isotope	Element of Interest
27	Aluminum
121,123	Antimony
75	Arsenic
135,137	Barium
9	Beryllium
106,108,111,114	Cadmium
52,53	Chromium
59	Cobalt
63,65	Copper
206,207,208	Lead
55	Manganese
95,97,98	Molybdenum
60,62	Nickel
77,82	Selenium
107,109	Silver
203,205	Thallium
232	Thorium
238	Uranium
51	Vanadium
66,67,68	Zinc
83	Krypton
99	Ruthenium
105	Palladium
118	Tin

NOTE: Isotopes recommended for analytical determination are underlined.

TABLE 2 (Continued).

MATRIX MOLECULAR IONS

MATRIX OXIDES*

Molecular Ion	Masses	Element Interference
TiO	62-66	Ni,Cu,Zn
ZrO	106-112	Ag,Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

TABLE 2: COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

BACKGROUND MOLECULAR IONS		
Molecular Ion	Mass	Element Interference[a]
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H^+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

[a] method elements or internal standards affected by the molecular ions.

3. Houk, R.S., Mass Spectrometry of Inductively Coupled Plasmas. *Anal. Chem.* 58 97A-105A, 1986.

4. Thompson, J.J. and R. S. Houk. A Study of Internal Standardization in Inductively Coupled Plasma-Mass Spectrometry. *Appl. Spec.* 41 801-806, 1987.

5. Carcinogens - Working With Carcinogens, Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977. Available from the National Technical Information Service (NTIS) as PB-277256.

6. OSHA Safety and Health Standards, General Industry, (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

7. Safety in Academic Chemistry Laboratories, American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

8. Proposed OSHA Safety and Health Standards, Laboratories, Occupational Safety and Health Administration, Federal Register, July 24, 1986.

9. American Society for Testing and Materials. Standard Specification for Reagent Water, D1193-77. Annual Book of ASTM Standards, Vol. 11.01. Philadelphia, PA, 1991.

10. Code of Federal Regulations 40, Ch. 1, Pt. 136 Appendix B.

11. Longbottom, J.E. et. al. Determination of Trace Elements in Water by Inductively Coupled Plasma-Mass Spectrometry: Collaborative Study, Journal of AOAC International 77 1004-1023, 1994.

12. Hinners, T.A., Interferences in ICP-MS by Bromine Species. Winter Conference on Plasma Spectrochemistry, San Diego, CA, January, 10-15, 1994.

both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

12.8 The QC data obtained during the analyses provide an indication of the quality of the sample data and should be provided with the sample results.

13.0 METHOD PERFORMANCE

13.1 Instrument operating conditions used for single laboratory testing of the method are summarized in Table 6. Total recoverable digestion and "direct analysis" MDLs determined using the procedure described in Sect. 9.2.4, are listed in Table 7.

13.2 Data obtained from single laboratory testing of the method are summarized in Table 9 for five water samples representing drinking water, surface water, ground water and waste effluent. Samples were prepared using the procedure described in Sect. 11.2. For each matrix, five replicates were analyzed and the average of the replicates used for determining the sample background concentration for each element. Two further pairs of duplicates were fortified at different concentration levels. For each method element, the sample background concentration, mean percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate fortified samples are listed in Table 8.

13.3 Data obtained from single laboratory testing of the method are summarized in Table 10 for three solid samples consisting of SRM 1645 River Sediment, EPA Hazardous Soil and EPA Electroplating Sludge. Samples were prepared using the procedure described in Sect. 11.3. For each method element, the sample background concentration, mean percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate fortified samples were determined as for Sect. 13.2.

13.4 Data obtained from single laboratory testing of the method for drinking water analysis using the "direct analysis" procedure (Sect. 11.2.1) are given in Table 11. Three drinking water samples of varying hardness collected from Regions 4, 6, and 10 were fortified to contain 1 µg/L of all metal primary contaminants, except selenium, which was added to a concentration of 20 µg/L. For each matrix, four replicate aliquots were analyzed to determine the sample background concentration of each analyte and four fortified aliquots were analyzed to determine mean percent recovery in each matrix. Listed in the Table 11 are the average mean percent recovery of each analyte in the three matrices and the standard deviation of the mean percent recoveries.

13.5 Listed in Table 12 are the regression equations for precision and bias developed from the joint USEPA/Association of Official Analytical Chemists (AOAC) multilaboratory validation study conducted on this

11.4.2 Initiate instrument operating configuration. Tune and calibrate the instrument for the analytes of interest (Sect. 10.0).

11.4.3 Establish instrument software run procedures for quantitative analysis. For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Use the average of the integrations for data reporting.

11.4.4 All masses which might affect data quality must be monitored during the analytical run. As a minimum, those masses prescribed in Table 4 must be monitored in the same scan as is used for the collection of the data. This information should be used to correct the data for identified interferences.

11.4.5 During the analysis of samples, the laboratory must comply with the required quality control described in Sections 9.3 and 9.4. Only for the determination of dissolved analytes or the "direct analysis" of drinking water with turbidity of < 1 NTU is the sample digestion step of the LRB, LFB, and LFM not required.

11.4.6 The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of one minute (Sect. 4.1.5). Samples should be aspirated for 30 sec prior to the collection of data.

11.4.7 Samples having concentrations higher than the established linear dynamic range should be diluted into range and reanalyzed. The sample should first be analyzed for the trace elements in the sample, protecting the detector from the high concentration elements, if necessary, by the selection of appropriate scanning windows. The sample should then be diluted for the determination of the remaining elements. Alternatively, the dynamic range may be adjusted by selecting an alternative isotope of lower natural abundance, provided quality control data for that isotope have been established. The dynamic range must not be adjusted by altering instrument conditions to an uncharacterized state.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Elemental equations recommended for sample data calculations are listed in Table 5. Sample data should be reported in units of µg/L for aqueous samples or mg/kg dry weight for solid samples. Do not report element concentrations below the determined MDL.

12.2 For data values less than ten, two significant figures should be used for reporting element concentrations. For data values greater than or equal to ten, three significant figures should be used.

12.3 For aqueous samples prepared by total recoverable procedure (Sect. 11.2), multiply solution concentrations by the dilution factor 1.25. If additional dilutions were made to any samples or an aqueous sample

the sample may be filtered for their removal prior to analysis. However, care should be exercised to avoid potential contamination from filtration.)

11.2.8 Prior to analysis, adjust the chloride concentration by pipetting 20 mL of the prepared solution into a 50-mL volumetric flask, dilute to volume with reagent water and mix. (If the dissolved solids in this solution are > 0.2%, additional dilution may be required to prevent clogging of the extraction and/or skimmer cones. If the direct addition procedure (Method A, Sect. 10.3) is being used, add internal standards and mix. The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

11.3 Solid Sample Preparation - Total Recoverable Analytes

11.3.1 For the determination of total recoverable analytes in solid samples, mix the sample thoroughly and transfer a portion (> 20 g) to tared weighing dish, weigh the sample and record the wet weight (WW). (For samples with < 35% moisture a 20 g portion is sufficient. For samples with moisture > 35% a larger aliquot 50-100 g is required.) Dry the sample to a constant weight at 60°C and record the dry weight (DW) for calculation of percent solids (Sect. 12.6). (The sample is dried at 60°C to prevent the loss of mercury and other possible volatile metallic compounds, to facilitate sieving, and to ready the sample for grinding.)

11.3.2 To achieve homogeneity, sieve the dried sample using a 5-mesh polypropylene sieve and grind in a mortar and pestle. (The sieve, mortar and pestle should be cleaned between samples.) From the dried, ground material weigh accurately a representative 1.0 ± 0.01 g aliquot (W) of the sample and transfer to a 250-mL Phillips beaker for acid extraction.

11.3.3 To the beaker add 4 mL of (1+1) HNO_3 and 10 mL of (1+4) HCl. Cover the lip of the beaker with a watch glass. Place the beaker on a hot plate for reflux extraction of the analytes. The hot plate should be located in a fume hood and previously adjusted to provide a reflux temperature of approximately 95°C. (See the following note.)

NOTE: For proper heating adjust the temperature control of the hot plate such that an uncovered Griffin beaker containing 50 mL of water placed in the center of the hot plate can be maintained at a temperature approximately but no higher than 85°C. (Once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95°C.) Also, a block digester capable of maintaining a temperature of 95°C

Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

10.4 Calibration - Prior to initial calibration, set up proper instrument software routines for quantitative analysis. The instrument must be calibrated using one of the internal standard routines (Method A or B) described in Section 10.3. The instrument must be calibrated for the analytes to be determined using the calibration blank (Sect. 7.6.1) and calibration standards A and B (Sect. 7.4.1) prepared at one or more concentration levels. A minimum of three replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

10.5 The rinse blank should be used to flush the system between solution changes for blanks, standards and samples. Allow sufficient rinse time to remove traces of the previous sample (Sect. 4.1.5). Solutions should be aspirated for 30 sec prior to the acquisition of data to allow equilibrium to be established.

11.0 PROCEDURE

11.1 Aqueous Sample Preparation - Dissolved Analytes

11.1.1 For the determination of dissolved analytes in ground and surface waters, pipet an aliquot ($\geq$ 20 mL) of the filtered, acid preserved sample into a 50-mL polypropylene centrifuge tube. Add an appropriate volume of (1+1) nitric acid to adjust the acid concentration of the aliquot to approximate a 1% (v/v) nitric acid solution (e.g., add 0.4 mL (1+1) HNO_3 to a 20 mL aliquot of sample). If the direct addition procedure (Method A, Sect. 10.3) is being used, add internal standards, cap the tube and mix. The sample is now ready for analysis (Sect. 1.2). Allowance for sample dilution should be made in the calculations.

NOTE: If a precipitate is formed during acidification, transport, or storage, the sample aliquot must be treated using the procedure in Section 11.2 prior to analysis.

11.2 Aqueous Sample Preparation - Total Recoverable Analytes

11.2.1 For the "direct analysis" of total recoverable analytes in drinking water samples containing turbidity < 1 NTU, treat an unfiltered acid preserved sample aliquot using the sample preparation procedure described in Section 11.1.1 while making allowance for sample dilution in the data calculation. For the determination of total recoverable analytes in all other aqueous samples or for preconcentrating drinking water samples prior to analysis follow the procedure given in Sections 11.2.2 through 11.2.8.

9.4.3 Calculate the percent recovery for each analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range of 70-130%. Recovery calculations are not required if the concentration of the analyte added is less than 30% of the sample background concentration. Percent recovery may be calculated in units appropriate to the matrix, using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where: R = percent recovery.
C_s = fortified sample concentration.
C = sample background concentration.
s = concentration equivalent of analyte added to fortify the sample.

9.4.4 If recovery of any analyte falls outside the designated range and laboratory performance for that analyte is shown to be in control (Sect. 9.3), the recovery problem encountered with the fortified sample is judged to be matrix related, not system related. The data user should be informed that the result for that analyte in the unfortified sample is suspect due to either the heterogeneous nature of the sample or an uncorrected matrix effect.

9.4.5 Internal standards responses - The analyst is expected to monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the internal standards responses against each other should also be monitored routinely. This information may be used to detect potential problems caused by mass dependent drift, errors incurred in adding the internal standards or increases in the concentrations of individual internal standards caused by background contributions from the sample. The absolute response of any one internal standard must not deviate more than 60-125% of the original response in the calibration blank. If deviations greater than these are observed, flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor of two, add the internal standards and reanalyze. If after flushing the response of the internal standards in the calibration blank are out of limits, terminate the analysis and determine the cause of the drift. Possible causes of drift may be a partially blocked sampling cone or a change in the tuning condition of the instrument.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Operating conditions - Because of the diversity of instrument hardware, no detailed instrument operating conditions are provided.

1.2). MDLs should be determined annually, when a new operator begins work or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

9.3 Assessing Laboratory Performance (mandatory)

9.3.1 Laboratory reagent blank (LRB) - The laboratory must analyze at least one LRB (Sect. 7.6.2) with each batch of 20 or fewer of samples of the same matrix. LRB data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing. LRB values that exceed the MDL indicate laboratory or reagent contamination should be suspected. When LRB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained.

9.3.2 Laboratory fortified blank (LFB) - The laboratory must analyze at least one LFB (Sect. 7.9) with each batch of samples. Calculate accuracy as percent recovery using the following equation:

$$R = \frac{LFB - LRB}{s} \times 100$$

where: R = percent recovery.
LFB = laboratory fortified blank.
LRB = laboratory reagent blank.
s = concentration equivalent of analyte added to fortify the LRB solution.

If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.3 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 85-115% (Sect.9.3.2). When sufficient internal performance data become available (usually a minimum of twenty to thirty analyses), optional control limits can be developed from the mean percent recovery (x) and the standard deviation (S) of the mean percent recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x - 3S

be > 2, more acid must be added and the sample held for sixteen hours until verified to be pH < 2. See Section 8.1.

NOTE: When the nature of the sample is either unknown or known to be hazardous, acidification should be done in a fume hood. See Section 5.2.

8.4 Solid samples require no preservation prior to analysis other than storage at 4°C. There is no established holding time limitation for solid samples.

8.5 For aqueous samples, a field blank should be prepared and analyzed as required by the data user. Use the same container and acid as used in sample collection.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and calibration solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

9.2 Initial Demonstration of Performance (mandatory)

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges and analysis of quality control samples) and laboratory performance (determination of method detection limits) prior to analyses conducted by this method.

9.2.2 Linear calibration ranges - Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. The upper LDR limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

9.2.3 Quality control sample (QCS) - When beginning the use of this method, on a quarterly basis or as required to meet data-

should be verified initially using a quality control sample (Sect. 7.8).

7.5 Internal Standards Stock Solution - 1 mL = 100 μg. Dilute 10 mL of scandium, yttrium, indium, terbium and bismuth stock standards (Sect. 7.3) to 100 mL with reagent water, and store in a FEP bottle. Use this solution concentrate for addition to blanks, calibration standards and samples, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump (Method B, Sect. 10.3).

NOTE: If mercury is to be determined by the "direct analysis" procedure, add an aliquot of the gold stock standard (Sect. 7.3.11) to the internal standard solution sufficient to provide a concentration of 100 μg/L in final the dilution of all blanks, calibration standards, and samples.

7.6 Blanks - Three types of blanks are required for this method. A calibration blank is used to establish the analytical calibration curve, the laboratory reagent blank is used to assess possible contamination from the sample preparation procedure and to assess spectral background and the rinse blank is used to flush the instrument between samples in order to reduce memory interferences.

7.6.1 Calibration blank - Consists of 1% (v/v) nitric acid in reagent grade water. If the direct addition procedure (Method A, Sect. 10.3) is being used, add internal standards.

7.6.2 Laboratory reagent blank (LRB) - Must contain all the reagents in the same volumes as used in processing the samples. The LRB must be carried through the same entire preparation scheme as the samples including digestion, when applicable. If the direct addition procedure (Method A, Sect. 10.3) is being used, add internal standards to the solution after preparation is complete.

7.6.3 Rinse blank - Consists of 2% (v/v) nitric acid in reagent grade water.

NOTE: If mercury is to be determined by the "direct analysis" procedure, add gold (Sect. 7.3.11) to the rinse blank to a concentration of 100 μg/L.

7.7 Tuning Solution - This solution is used for instrument tuning and mass calibration prior to analysis. The solution is prepared by mixing beryllium, magnesium, cobalt, indium and lead stock solutions (Sect. 7.3) in 1% (v/v) nitric acid to produce a concentration of 100 μg/L of each element. Internal standards are not added to this solution. (Depending on the sensitivity of the instrument, this solution may need to be diluted 10 fold.)

7.8 Quality Control Sample (QCS) - The QCS should be obtained from a source outside the laboratory. The concentration of the QCS solution

7.3.16 Mercury solution, stock, 1 mL = 1000 μg Hg: DO NOT DRY. **CAUTION:** highly toxic element. Dissolve 0.1354 g $HgCl_2$ in reagent water. Add 5.0 mL concentrated HNO_3 and dilute to 100 mL with reagent water.

7.3.17 Molybdenum solution, stock 1 mL = 1000 μg Mo: Dissolve 0.1500 g MoO_3 in a solution mixture of 10 mL reagent grade water and 1 mL conc. ammonium hydroxide., heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.3.18 Nickel solution, stock 1 mL = 1000 μg Ni: Dissolve 0.100 g nickel powder in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.3.19 Scandium solution, stock 1 mL = 1000 μg Sc: Dissolve 0.1534 g Sc_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.3.20 Selenium solution, stock 1 mL = 1000 μg Se: Dissolve 0.1405 g SeO_2 in 20 mL ASTM type I water. Dilute to 100 mL with reagent grade water.

7.3.21 Silver solution, stock 1 mL = 1000 μg Ag: Dissolve 0.100 g silver metal in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water. Store in dark container.

7.3.22 Terbium solution, stock 1 mL = 1000 μg Tb: Dissolve 0.1176 g Tb_4O_7 in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.3.23 Thallium solution, stock 1 mL = 1000 μg Tl: Dissolve 0.1303 g $TlNO_3$ in a solution mixture of 10 mL reagent grade water and 1 mL conc. nitric acid. Dilute to 100 mL with reagent grade water.

7.3.24 Thorium solution, stock 1 mL = 1000 μg Th: Dissolve 0.2380 g $Th(NO_3)_4.4H_2O$ (DO NOT DRY) in 20 mL reagent grade water. Dilute to 100 mL with reagent grade water.

7.3.25 Uranium solution, stock 1 mL = 1000 μg U: Dissolve 0.2110 g $UO_2(NO_3)_2.6H_2O$ (DO NOT DRY) in 20 mL reagent grade water and dilute to 100 mL with reagent grade water.

7.3.26 Vanadium solution, stock 1 mL = 1000 μg V: Pickle vanadium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.3.27 Yttrium solution, stock 1 mL = 1000 μg Y: Dissolve 0.1270 g Y_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent grade water.

7.2 Reagent water - All references to reagent grade water in this method refer to ASTM type I water (ASTM D1193).[9] Suitable water may be prepared by passing distilled water through a mixed bed of anion and cation exchange resins.

7.3 Standard Stock Solutions - Stock standards may be purchased from a reputable commercial source or prepared from ultra high-purity grade chemicals or metals (99.99 - 99.999% pure). All salts should be dried for 1 h at 105°C, unless otherwise specified. Stock solutions should be stored in FEP bottles. Replace stock standards when succeeding dilutions for preparation of the multielement stock standards can not be verified.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

The following procedures may be used for preparing standard stock solutions:

NOTE: Some metals, particularly those which form surface oxides require cleaning prior to being weighed. This may be achieved by pickling the surface of the metal in acid. An amount in excess of the desired weight should be pickled repeatedly, rinsed with water, dried and weighed until the desired weight is achieved.

7.3.1 Aluminum solution, stock 1 mL = 1000 μg Al: Pickle aluminum metal in warm (1+1) HCl to an exact weight of 0.100 g. Dissolve in 10 mL conc. HCl and 2 mL conc. nitric acid, heating to effect solution. Continue heating until volume is reduced to 4 mL. Cool and add 4 mL reagent grade water. Heat until the volume is reduced to 2 mL. Cool and dilute to 100 mL with reagent grade water.

7.3.2 Antimony solution, stock 1 mL = 1000 μg Sb: Dissolve 0.100 g antimony powder in 2 mL (1+1) nitric acid and 0.5 mL conc. hydrochloric acid, heating to effect solution. Cool, add 20 mL reagent grade water and 0.15 g tartaric acid. Warm the solution to dissolve the white precipitate. Cool and dilute to 100 mL with reagent grade water.

7.3.3 Arsenic solution, stock 1 mL = 1000 μg As: Dissolve 0.1320 g As_2O_3 in a mixture of 50 mL reagent grade water and 1 mL conc. ammonium hydroxide. Heat gently to dissolve. Cool and acidify the solution with 2 mL conc. nitric acid. Dilute to 100 mL with reagent grade water.

7.3.4 Barium solution, stock 1 mL = 1000 μg Ba: Dissolve 0.1437 g $BaCO_3$ in a solution mixture of 10 mL reagent grade water and 2 mL conc. nitric acid. Heat and stir to effect solution and degassing. Dilute to 100 mL with reagent grade water.

concentrations of elements beyond the linear range of the instrument and with isotopes falling within scanning windows should be diluted prior to analysis.

6.2 Analytical balance, with capability to measure to 0.1 mg, for use in weighing solids, for preparing standards, and for determining dissolved solids in digests or extracts.

6.3 A temperature adjustable hot plate capable of maintaining a temperature of 95°C.

6.4 (optional) A temperature adjustable block digester capable of maintaining a temperature of 95°C and equipped with 250-mL constricted digestion tubes.

6.5 (optional) A steel cabinet centrifuge with guard bowl, electric timer and brake.

6.6 A gravity convection drying oven with thermostatic control capable of maintaining 105°C ± 5°C.

6.7 (optional) An air displacement pipetter capable of delivering volumes ranging from 0.1 to 2500 µL with an assortment of high quality disposable pipet tips.

6.8 Mortar and pestle, ceramic or nonmetallic material.

6.9 Polypropylene sieve, 5-mesh (4 mm opening).

6.10 Labware - For determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) should be sufficiently clean for the task objectives. Several procedures found to provide clean labware include soaking overnight and thoroughly washing with laboratory-grade detergent and water, rinsing with tap water, and soaking for four hours or more in 20% (V/V) nitric acid or a mixture of dilute nitric and hydrochloric acid (1+2+9), followed by rinsing with reagent grade water and storing clean.

NOTE: Chromic acid must not be used for cleaning glassware.

6.10.1 Glassware - Volumetric flasks, graduated cylinders, funnels and centrifuge tubes (glass and/or metal free plastic).

6.10.2 Assorted calibrated pipettes.

extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended[3] to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects.[4] Internal standards ideally should have similar analytical behavior to the elements being determined.

4.1.5 Memory interferences - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples (Sect. 7.6.3). The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be reanalyzed after a long rinse period. In the determination of mercury, which suffers from severe memory effects, the addition of 100 µg/L gold will effectively rinse 5 µg/L mercury in approximately 2 minutes. Higher concentrations will require a longer rinse time.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.[5,8] A reference file of material data handling sheets should also be available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever

determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus (Sects. 7.6.2 & 9.3.1).

3.11 Linear Dynamic Range (LDR) - The concentration range over which the instrument response to an analyte is linear (Sect. 9.2.2).

3.12 Method Detection Limit (MDL) - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero (Sect. 9.2.4 and Table 7).

3.13 Quality Control Sample (QCS) - A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance (Sects. 7.8 & 9.2.3).

3.14 Solid Sample - For the purpose of this method, a sample taken from material classified as either soil, sediment or sludge.

3.15 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source (Sect. 7.3).

3.16 Total Recoverable Analyte - The concentration of analyte determined either by "direct analysis" of an unfiltered acid preserved drinking water sample with turbidity of < 1 NTU (Sect. 11.2.1), or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method (Sects. 11.2 & 11.3).

3.17 Tuning Solution - A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses (Sect. 7.7).

3.18 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm runoff, industrial or domestic wastewater.

4.0 INTERFERENCES

4.1 Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

4.1.1 Isobaric elemental interferences - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table 4), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric

13.2. Common isobaric interferences are corrected using equations equivalent to those listed in EPA Method 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA Method 200.8 for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3. Data Review and Reporting

13.3.1. Production

Data is reviewed by the ICP-MS operator and a report is generated. A senior spectroscopist performs a final review of the data and associated report. The data is then placed in the holding file until all analyses are complete. When the work order is complete, a final review is performed and the data is delivered to the project management department.

13.3.2. Non-production

Method Development/Research and Development studies are performed under the direction of a senior spectroscopist. All associated data is scrutinized by the senior spectroscopist. Original raw data and associated records are archived in the analytical project file.

14. TRAINING

14.1. A minimum of two senior level spectroscopists are to be maintained on staff at all times. Senior spectroscopists are defined as individuals with a minimum of ten years combined education and experience in, or related to atomic spectroscopy. Of those ten years, a minimum of two years of ICP-MS experience is required.

14.2. To maintain expertise in current technology, senior staff members are required to attend at least one technical seminar per year containing significant information relevant to ICP-MS. All technical staff are encouraged to attend one technical seminar per year.

14.3. In addition to the technical seminars, senior spectroscopists are required to complete a one week training session offered by the instrument manufacturer.

14.4. On-the-job-training occurs daily with the senior spectroscopists providing direction to new operators. The physical operation of the equipment is relatively simple. The data reduction and troubleshooting requires extensive experience which can only be gained by hands-on operation of the instrument and assisted evaluation of raw data.

15. REFERENCES

15.1. USEPA, Contract Laboratory Program, SOW #ILM04.0

15.2. Thermo Jarrell Ash ICAP61 Manual

15.3. USEPA, Methods for Determination of Metals in Environmental Samples, Method 200.8, Revision 5.4, May 1994.

STANDARD OPERATING PROCEDURE

PARTICLE SIZE/PUGET SOUND ESTUARY PROGRAM

GEN-PSP
Revision 1
August 23, 1999

Approved By:

Supervisor	8/23/99 Date
QA Manager	8-23-99 Date
Laboratory Manager	8-23-99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
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PARTICLE SIZE/PUGET SOUND ESTUARY PROGRAM

1. SCOPE AND APPLICATION

1.1. Applicable Matrices

This method is designed to determine the fraction of pre-determined sizes of particles in sediments.

1.2. Range

Particle size can be characterized in a wide range of detail. The grossest divisions that generally are considered useful for characterizing particle size distributions are percentages of gravel, sand, silt, and clay. However, each of these size fractions can be subdivided further so that additional characteristics of the size distribution (e.g., mean diameter, skewness, kurtosis) can be determined.

1.3. Method Detection Limits

Detection limits are determined from accuracy of analytical balances. Samples are weighed to the nearest 0.01g and results are reported to the nearest 0.01 percent.

2. METHOD SUMMARY

2.1. Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological variables, it can be used to normalize chemical concentrations according to sediment characteristics and to account for some of the variability found in biological assemblages. Particle size is also an important variable for marine engineering purposes. In addition to Plumb (1981), a variety of other references discuss the uses and measurement of particle size (e.g., Krumbein and Pettijohn 1938; Folk 1968; Buchanan 1984).

2.2. Particle size determinations can either include or exclude organic material. If organic material is removed prior to analysis, the "true" (i.e., primarily inorganic) particle size distribution is determined. If organic material is included in the analysis, the "apparent" (i.e., organic plus inorganic) particle size distribution is determined. Because true and apparent distributions may differ, detailed comparisons between samples analyzed by these different methods are questionable. It is therefore desirable that all samples within each study (at a minimum) and among different studies (if possible) be analyzed using only one of these two methods.

3. INTERFERENCES

Depending on the required particle size distribution, organic material can be an interference.

4. SAFETY

Personal protective equipment will include safety glasses (with side shields), gloves, and a lab coat. Follow normal precautions as per the CAS Safety Manual.

5. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

5.1. Collection

Samples can be collected in glass or plastic containers. A minimum sample size of 100-150g is recommended. If unrepresentative material is to be removed from the sample, it should be removed in the field under the supervision of the chief scientist and noted on the field log sheet.

5.2. Processing

Samples should be stored at 4 ± 2 °C, and can be held for up to 6 months before analysis. Samples must not be frozen or dried prior to analysis, as either process may change the particle size distribution.

6. APPARATUS AND EQUIPMENT

6.1. Sieve shaker - Ro-Tap or equivalent

6.2. Drying oven

6.3. Constant temperature bath

6.4. Analytical balance - 0.1mg accuracy

6.5. Desiccator

6.6. Clock - with second hand

6.7. Standard sieves - Appropriate mesh sizes

6.8. Sieve pan and top, sieve brush

6.9. Funnel

6.10. Graduated cylinders

6.11. 50-mL beakers

6.12. 20-mL pipets

6.13. Water pique or squirt bottle

6.14. Glossy paper

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Dispersant - 1 percent sodium hexamethaphosphate = 1 percent commercially available Calgon

7.2. Distilled water

8. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9. PREVENTATIVE MAINTENANCE

No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

10. PROCEDURE

10.1. Refer to Puget Sound Estuary Program Protocols for Conventional Sediment Variables, March 1986. Exceptions to this procedure are outlined below.

10.2. Sample Preparation

Unless specifically asked for, organic oxidation is not performed and the apparent particle size distribution is determined.

10.3. Analysis

10.3.1. Gravel-sand fraction - coarse fraction is washed into a preweighed 150ml beaker instead of a 50-ml beaker.

10.3.2. Silt-clay fraction - after sample is brought to 1L and mixed, it is left on the counter and the temperature is tracked with a thermometer in an erlenmeyer flask with DI water. The temperature does not usually vary by more than ± 2°C.

10.3.3. Withdrawal times for pipet analysis as a function of particle size and water temperature are given in Table 1.

11. QA/QC REQUIREMENTS

11.1. It is critical that each sample be homogenized thoroughly in the laboratory before a subsample is taken for analysis. Laboratory homogenization should be conducted even if samples were homogenized in the field.

11.2. After dry-sieving a sample, all material must be removed from the sieve. This can be accomplished by tapping the rim of the sieve evenly on a hard surface and by brushing the screen.

11.3. The total amount of fine-grained material used for pipet analysis should be 5-25g. If more material is used, particles may interfere with each other during settling and the possibility of flocculation may be enhanced. If less material is used, the experimental error in weighing becomes large relative to the sample size.

11.4. Before pipet extractions can be made, the sample must be homogenized thoroughly within the settling cylinder. Once the pipet analysis begins, the settling cylinders must not be disturbed, as this will alter particle settling velocities. Care must be taken to disturb the sample as little as possible when pipet extractions are made.

11.5. After a pipet extract has been transferred to a drying beaker, any sample adhering to the inside of the pipet must be removed. This can be accomplished by drawing 20mL of distilled water into the pipet and adding this rinse water to the drying beaker.

11.6. Dried samples should be cooled in a desiccator and held there until they are weighed. If a desiccator is not used, the sediment will accumulate ambient moisture and the sample

weight will be overestimated. A color-indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically, and, if necessary, the ground glass rims should be greased or the "0" rings should be replaced.

11.7. It is recommended that triplicate analyses be conducted on one of every 20 samples, or on one sample per batch if less than 20 samples are analyzed. It is also recommended that the analytical balance, drying oven, and temperature bath be inspected daily and calibrated at least once per week.

12. DATA REDUCTION, REVIEW, AND REPORTING

12.1. Calculations

12.1.1. The total weight of a phi-size interval in the 1-L graduated cylinder is determined as follows:

Phi weight (g dry weight) = 50[(A-C)-(B-C)]

Where:
A = weight (g) of residue in a 20-mL aliquot for a given phi-size boundary
B = weight (g) residue in a 20-mL aliquot for the next larger phi-size boundary
C = mean weight (g) of dispersant in a 20-mL aliquot.

12.2. The data is entered into a spread sheet and results determined using the appropriate equations.

12.3. Reporting and review

12.3.1. The weight of each sediment fraction should be reported to the nearest 0.0001g dry weight. The laboratory should report the results of all samples analyzed (including QA replicates) and should note any problems that may have influenced data quality.

12.3.2. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified for samples (above). These results are then used to calculate QC determinations

12.3.3. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in

the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS. Once the results are transferred, the report is reviewed.

12.3.4. Refer to the SOP for Laboratory Data Review Process for general instructions for data review.

13. REFERENCES

Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, January, 1996

Procedures for Handling and Chemical Analysis of Sediment and Water Samples, R.H. Plumb, prepared for USEPA and Army Corps of Engineers, May, 1981.

Table 1

Withdrawal Times for Pipet Analysis as a Function of Particle Size and Water Temperature [a,b]

Micron μm	Diameter finer than (phi)	Diameter finer than (μm)	Withdrawal depth (cm)	Elapsed time for withdrawal of sample in hours (h), minutes (m), and seconds(s).						
				18°C	19°C	20°C	21°C	22°C	23°C	24°C
62.5	4.0	62.5 - 3.9	20	20s	20s	20s	20s	20s	20s	20s
3.9	8.0[c]	3.9	10	2h8m	2h5m	2h2m	1h59m	1h56m	1h53m	1h51m

a) Modified from Plumb (1981).
b) It is critical that temperature be held constant during the pipet analysis.
c) Breakpoint between silt and clay.

STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM D4129-82 M
Revision 2
August 31, 1999

Approved By:

Supervisor	8/31/99 Date
QA Manager	8-31-99 Date
Laboratory Manager	9-1-99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
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CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-88, modified for soil and sediment matrices. Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. INTERFERENCES

3.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

3.2. Volatile organics may be lost in the decarbonization process.

4. SAFETY

4.1. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

4.2. Avoid skin contact with all chemicals.

4.3. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

5. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4°C.

6. APPARATUS AND EQUIPMENT

6.1. Induction furnace, Coulometrics Incorporated.
6.2. Analytical balance, 0.1mg accuracy.
6.3. Desiccator.
6.4. Platinum combustion boats.
6.5. Ten percent hydrochloric acid (HCl).
6.6. Sample scoop.
6.7. Porcelain dishes.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Standards

Urea - 20% carbon. Use 10 µg.

Benzoic Acid - 68.8% carbon. Use 5 µg. Alternatively, a purchased standard of a known TOC value can be used (ERA #542, TOC/TKN/T. Phos/Ammonia in Soil).

7.2. Reagents

7.2.1. Hydrochloric acid, 10%.

7.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

7.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

7.2.4. Manganese dioxide. Gas scrubber solution.

7.2.5. Potassium Hydroxide. Gas scrubber solution.

7.2.6. Potassium Iodide. Anode chemical.

7.2.7. Magnesium Perchlorate desiccant

7.3. Consumables

Platinum boats and miscellaneous glassware, specifically glass ladles.

8. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9. PREVENTATIVE MAINTENANCE

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Combustion Column	as needed
Post Combustion Column	as needed

10. PROCEDURE

10.1. Sample Preparation.

10.1.1. Turn furnace on to #6 (≈1000°C). Allow furnace to warm-up for about 2 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

10.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

10.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

10.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10% HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

10.2. Apparatus Preparation.

10.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

10.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

10.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

10.2.4. Place cell in coulometer cell holder.

10.2.5. Turn on detector lamp and stir plate. (Power on)

10.2.6. Rotate cell until maximum transmittance is obtained. 60% or better. 100% transmittance is difficult to obtain due to the color of the carbon cathode solution.

10.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

10.2.8. Change Magnesium Perchlorate desiccant after each bracket.

10.2.9. The instrument is now ready to run.

10.3. Calibration and Standardization.

10.3.1. Burn both ladles for five minutes each to remove any residual TOC.

10.3.2. Establish baseline.

10.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

10.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

10.4. Analysis.

10.4.1. Place one platinum boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

10.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

10.4.3. Load the other ladle with the next platinum boat. Remove the ladle in use from the inlet port and insert the next ladle.

10.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

11. QA/QC REQUIREMENTS

11.1. Refer to the SOP for Analytical Batches and Analytical Sequences for batching instructions. Project-specific batching protocols may also be required.

11.2. QC Samples Required.

11.2.1. LCS (Benzoic Acid) - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze ≈5mg. The acceptance criteria for the LCS is 58.5% - 79.1% carbon for benzoic acid. Alternatively, ERA standard (see 7.1) may be used. The acceptance criteria for this LCS is ± 15% of the true value.

11.2.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be <0.05% carbon.

11.2.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Urea CCV must be 18.0% - 22.0% carbon.

11.2.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

11.2.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

11.2.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

11.2.7. See Table 1 for a summary of accetance criteria and corrective actions.

12. DATA REDUCTION AND REPORTING

12.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ -\ baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

12.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

12.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

12.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

12.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

12.7. Reporting

12.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

12.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

12.7.3. Report all results to three significant figures.

12.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

13. REFERENCES

13.1. Coulometrics Inc. Instruction Manual, Model 5020.

13.2. EPA Method Modified 415.1.

13.3. *Total Organic Carbon*, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0

13.4. ASTM Method D4129-88

TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

APPENDIX I

BENCHSHEETS

Columbia Analytical Services, Inc.

vice Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

lysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

nple Number								
Sample Injected								
nple Reading, µg C								
eline Reading, µg C								
µg C								
arbon								

nple Number								
Sample Injected								
nple Reading, µg C								
eline Reading, µg C								
µg C								
arbon								

nple Number								
le Injected								
nple Reading, µg C								
eline Reading, µg C								
µg C								
arbon								

d Purge Time: 1 min. Reading Time: 5 min. TOC % = (Net Reading)(µg 0.1) / mg Sample Injected

/ Urea
her(lot #0953513) TV = 20.0% C
: TOCS/1-10B % Rec =

LCS ERA
cat# 542 (lot# 01125) TV = 0.62%C
ID#: TOCS/1-10C % Rec =

nments: Baseline = 1) 2) 3) Avg. =

By:	Date:
eiwed By:	Date:

TOCSOIL.XLT

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
LCS				
MB				
CCV-1				
CCB-1				Avg
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

STANDARD OPERATING PROCEDURE

AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE

GEN-350.3
Revision 1
April 1, 1996

Approved By:

Supervisor	6/14/96 Date
QA Coordinator	6-14-96 Date
Laboratory Manager	6-14-96 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

AMMONIA AS NITORGEN BY ION SPECIFIC ELECTRODE

1. PURPOSE

An ammonia selective electrode is used to determine the concentration of ammonia in a KCl extract of a soil sample or of a water sample. Known additions are used for the quantitation of ammonia in the sample.

2. INTERFERENCES

Volatile amines act as a positive interference. Mercury interferes by forming a strong complex with ammonia. Samples that form a precipitate upon the addition of sodium hydroxide will block the pores on the gas-permeable membrane causing a negative interference.

3. APPARATUS/REAGENTS

Orion 920A Specific Ion Analyzer
Orion Ammonia Electrode
150 mL Beakers, Volumetric Flasks
Stir Bars
Stir Plate
10 N NaOH (400g/L)
1,000 mg/L NH_4Cl (3.819g NH_4Cl/L)
100 mg/L NH_4Cl (Dilute 10.0 mL of 1,000 mg/L NH_4Cl to 100 mL)
10 mg/L NH_4Cl (Dilute 1.0 mL of 1,000 mg/L NH_4Cl to 100 mL)

4. PROCEDURE

4.1 Initial Instrument Set Up

4.1.1 Push "mode".

4.1.2 Push "2nd" "Electrode ID".

4.1.3 If the NH_3 method is not in the screen, push "no" until 09 (NH_3) is on screen. Push "0", "9", "yes".

4.1.4 Push "mode" until the instrument is reading concentration.

4.1.5 Push "calibrate".

4.2 Instrument Calibration

4.2.1 Prepare 10 mg/L and 100 mg/L standard solutions from 1,000 mg/L NH_4Cl stock solution.

4.2.2 Put 50 mL of 100 mg/L standard into a 150 mL beaker and set on stir plate. Insert ammonia electrode into the solution. Add 1 mL of 10 N NaOH (pH > 10).

4.2.3 Enter the number of standards used for calibration "2". Enter the value of your first standard "100" (100 mg/L). Push "yes" after correct value is entered. Rinse off electrode.

4.2.4 Put 50 mL of 10 mg/L standard into a 150 mL beaker and set or stir plate. Insert ammonia electrode into the solution. Add 1 mL of 10 N NaOH (pH >10).

4.2.5 Enter the value of the standard "10" (10 mg/L). Push "yes" after correct value is entered. Rinse off electrode.

4.2.6 The slope will then be displayed.

4.3 Sample Analysis

4.3.1 Push "2nd" "Incr. Tech". Push "1" for known addition then "yes". Push "1" for single electrode then "yes". Enter the electrode slope determined during the instrument calibration step then push "yes".

4.3.2 Put 50 mL of sample into a 150 mL beaker. Insert ammonia electrode into the solution. Add 1 mL 10 N NaOH.

4.3.3 Enter sample volume (mL) "50" then push "yes". Enter total volume (mL) "50" then push "yes".

4.3.4 Push "2nd" "mv" to determine known addition spike level from the initial mv reading. Refer to Table I. Record this value. Push any key to exit.

4.3.5 Enter standard concentration of known addition spike then push "yes". Push "yes" when instrument asks "NH_3 in sample". Push "yes" to continue after stable.

4.3.6 Enter first standard volume (mL of known addition spike). Push "yes".

4.3.7 Add known addition spike then push "yes". When reading is stable push "yes". Sample result will be displayed in mg/L.

4.3.8 If sample concentration exceeds 100 ppm, dilute sample appropriately to bring within range.

TABLE 1
AMMONIA SPECIFIC ION ELECTRODE

(For 50 mL Sample Volumes)

Apparent Concentration (mg/L NH_3-N)	**Spike Volume** (mL)	**Concentration of Spiking Solution** (mg/L NH_3-N)
*0 - 0.3	0.5	10
0.3 - 1.0	2.5	10
0.5 - 4.0	0.5	100
2 - 10	2.5	100
5 - 40	0.5	1,000
20 - 100	2.5	1,000
50 - 100	5	1,000

* Pre-spike 0.5 mL of 10 mg/L spiking solution

5. QUALITY CONTROL

5.1 After the initial calibration of the instrument, the slope is determined and entered into the instrument. The slope will vary depending on the temperature (usually -57 to -59 mV).

5.2 An ICV (Initial Calibration Verification) is analyzed at the start of each analytical batch. The acceptance criteria is ±15%. If outside this limit, the cause of the problem needs to be solved before proceeding.

5.3 An ICB (Initial Calibration Blank) is analyzed following the ICV. It must be less than 0.05 mg/L.

5.4 A CCV (Continuing Calibration Verification) at 10 mg/L is analyzed after the ICB and every ten readings and at the end of the run. It must be ±15%.

5.5 A CCB (Continuing Calibration Blank) is analyzed after each CCV. It must be less than 0.05 mg/L.

5.6 Duplicates are analyzed at a frequency of 10%.

6. DOCUMENTATION

All data and corrective actions must be recorded into the bench records by the analyst.

7. REFERENCES

EPA Method 350.3.

STANDARD OPERATING PROCEDURE

SULFIDES, ACIDS VOLATILE

GEN-AVS
Revision 2
April 23, 1998

Approved By:

Supervisor	4/23/98 Date
QA Coordinator	4/23/98 Date
Laboratory Manager	5-6-98 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: WFR Date: 5-20-99
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NON-CONTROLLED COPY
Will Not Be Updated

SULFIDES, ACIDS VOLATILE

1. SCOPE AND APPLICATION

1.1. This procedure describes the determination of acid volatile sulfide (AVS) in soil, sediment, and other solids. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailability of metals in sediments. The procedure can be used to isolate those metals, referred to as Simultaneously Extracted Metals (SEM), solubilized during the acidification step. Analysis for these metals can then be done using the applicable metals determinative procedure.

1.2. Research has established that the relative amounts of SEM and AVS are important in the prediction of potential metal bioavailability; if the molar ratio of SEM for bivalent metals to AVS exceeds one, the toxic heavy metals in that sample are potentially bioavailable. This method uses the same conditions for release of both sulfide and metal from the sediment and thus provides a useful means of assessing the amount of metal associated with sulfide.

1.3. This method is capable of determining sulfides in the range of 0.5 mg/Kg to 30,000 mg/Kg dry weight. The Method Reporting Limit (MRL) 0.5 mg/Kg. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. Method detection limits are 0.01 mg/Kg on the spectrophotometer and 0.2 mg/Kg in the sample.

2. METHOD SUMMARY

2.1. The AVS in the sample is first converted to hydrogen sulfide (H_2S) by acidification with hydrochloric acid at room temperature. The H_2S is then purged from the sample and trapped in aqueous solution. The amount of sulfide that has been trapped is then determined. If SEM is to be determined, a portion of the aqueous solution is used to perform selected metals analyses.

2.2. The H_2S released by acidifying the sample is quantified using a colorimetric procedure. In the colorimetric method, the H_2S is trapped in sodium hydroxide. The sulfide reacts with N-N dimethyl-p-phenylenediamine to form methylene blue that is measured. This procedure is capable of determining AVS concentrations as low as 0.05 mg/Kg dry weight of sediment. By appropriate sample dilution, the maximum concentration of AVS which can be determined is at least 30,000 mg/Kg dry sediment.

3. DEFINITIONS

3.1. Acid Volatile Sulfide (AVS) - AVS is defined as sulfides that form hydrogen sulfide under the conditions of this test. This includes amorphous, moderately crystalline monosulfides, and other sulfides.

3.2. Method Detection Limit (MDL) - The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined as described in the SOP for The Determination of Method Detection Limits.

3.3. Stock Standard Solution - A concentrated solution of the analyte prepared in the laboratory using assayed reference compounds or purchased from a reputable commercial source.

3.4. Calibration Standards - Solutions prepared from the stock standard solution that is used to calibrate the method response with respect to analyte concentration.

4. INTERFERENCES

Contact with oxygen must be avoided in all stages from sampling to analysis. Consequently, the samples and standards should be protected from air from the time of sampling through the analytical procedure.

5. SAFETY

5.1. The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should be available to all personnel involved in the chemical analysis.

5.2. Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or well ventilated area. If a high concentration of hydrogen sulfide is detected in the air by the laboratory staff, sample handling procedures must be corrected. Exposure to H_2S in air must not exceed guidelines or regulations. The OSHA Permissible Exposure Limit (PEL) is 50ppm.

5.3. If samples originate from a highly contaminated area, appropriate sample handling procedures to minimize worker exposure must be followed.

6. SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1. Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

6.2. During storage, sulfide can be formed or lost due to biological activity, and sulfide can be lost by volatilization or oxidation. Metal speciation can change as a result of changes in sulfide concentration and as a result of other changes in the sample.

6.3. Samples should be collected in wide mouth jars with a minimum of air space above the sediment. If possible, the headspace should be filled with oxygen free nitrogen or argon. The jar lids must have Teflon or polyethylene liners.

6.4. Samples should be cooled to 4°C as soon as possible after collection. Samples maintained at 4°C have been found to have no significant loss of AVS for storage periods up to 2 weeks. Holding time for samples should not exceed 14 days.

7. APPARATUS AND EQUIPMENT

7.1. Glassware

7.1.1. AVS evolution and H_2S trapping - The Midi is typically used.

7.1.1.1. Midi setup: For each analytical train, use one 125 ml round bottom flask with a septum, 80 ml scrubber tower with impingers with non-fritted outlets. The round bottom flask contains the sediment and acid is introduced to it by a syringe inserted through the septum. The flasks are connected by tubing. Because sulfide may react with tubing and other surfaces, minimum lengths of tubing should be used as sleeves to connect the glass tubing.

7.1.1.2. Macro setup: For each analytical train, one 500 ml round bottom flask with a septum 250 ml scrubber towers with impingers with non-fritted outlets. The round bottom flask contains the sediment and acid is introduced to it by a syringe inserted through the septum. The flasks are connected by tubing. Because sulfide may react with tubing and other surfaces, minimum lengths of tubing should be used as sleeves to connect the glass tubing. The analyst should pay particular attention to the recovery of sulfide from standards in evaluating the apparatus.

7.1.1.3. The analyst should pay particular attention to the recovery of sulfide from standards in evaluating the apparatus used.

7.1.1.4. In all cases, the inlets are below the liquid level and the outlets are above the liquid levels. The apparatus is assembled as shown in Figure 1, and more than one analytical train can be connected to a single cylinder of nitrogen or argon if flow controllers are installed in the line.

7.2. Assorted calibrated pipettes and volumetric flasks.

7.3. Analytical balance - capable of weighing to 0.0001 g.

7.4. Magnetic stirrer, thermally insulated and Teflon-coated stirring bar.

7.5. Spectrophotometer - Capable of measuring absorbance at 670 nm.

7.6. Spectrophotometer cells.

8. REAGENTS AND CONSUMABLE MATERIALS

8.1. All water and reagents used in this method must be free of dissolved oxygen and sulfide. Prepare reagents fresh for each batch and use deaerated, deionized water by removing dissolved oxygen from the deionized water by vigorously bubbling with oxygen-free nitrogen or argon for approximately one hour. Deaerate reagents immediately before use by deaerating with oxygen-free nitrogen or argon.

8.2. Sulfide stock standard solution, approximately 0.05M or 50 µmoles/ml.

8.2.1. Weigh about 12 gram of $Na_2S \cdot 9H_2O$ and dissolve it in 1,000 ml of deionized water. Store in a brown bottle. To prevent air oxidation, the sulfide solution should be maintained under oxygen-free nitrogen or argon.

8.2.2. Standardize against thiosulfate solution.

8.2.2.1. Pipette 10.00 ml of 0.025N standard iodine solution into each of two 125-ml Erlenmeyer flasks.

8.2.2.2. Pipette 2.00 ml of sulfide stock standard solution into one flask. Pipette 2.00 ml of deionized water, as a laboratory reagent blank, into the other flask.

8.2.2.3. Add 5.00 ml of 6M HCl into each flask, swirl slightly, then cover and place in the dark for 5 minutes.

8.2.2.4. Titrate each with 0.025N thiosulfate until the yellow iodine color fades to a pale straw. Just before all the iodine has been titrated, add starch indicator dropwise to form a pale blue color. Continue the titration with the thiosulfate. The end point is reached when the blue color first disappears.

8.2.2.5. Calculate the sulfide concentration as follows:

$$Sulfide\ (mg/L) = \frac{(T_{blank} - T_{sample}) \times N_{S_2O_3^{2-}}}{V_{sample}} X \frac{1\ mole\ S^{2-}}{2\ equiv\ S^{2-}} x \frac{1000\ \mu moles}{1\ mmole} x \frac{32 mmole}{mg}$$

where T = volume of titrant used for the blank and sample (ml)
N = concentration of $S_2O^{2-}_3$ titrant
V = volume of sample used (ml), 2.00 ml recommended

8.3. Iodine Solution (approximately 0.025N)

8.3.1. Dissolve 25 g potassium iodine, KI, in 700 ml of reagent water in a 1 liter volumetric flask. Add 3.2 g iodine, I_2. allow to dissolve. Add 2 ml of 6N HCl and standardize against sodium thiosulfate as follows.

8.3.2. Titrate until amber color fades to yellow. Add starch indicator solution. Continue titration drop by drop until the blue color disappears.

8.3.3. Run in duplicate

8.3.4. Calculate the normality as follows:

$$\frac{mls\ S_2O_3 \times Normality\ of\ S_2O_3\ (0.02SN)}{mls\ Iodine\ titrated\ (5\ mls)}$$

8.4. Standard sodium thiosulfate solution (0.025N): Dissolve 6.205 ± 0.005 g $Na_2S_2O_3 \cdot 5H_2O$ in 500 ml reagent water. Add 18 ml of 0.5N NaOH, and dilute to 1 liter.

8.5. Starch indicator - Dissolve 1.0 gram soluble starch in 100 ml boiling deionized water.

8.6. Sulfide working standards - Prepare sulfide working standards using the sulfide stock standard solution in Section 8.2.1. The concentrations of the following standards will depend on the exact concentration of the sulfide stock standard determined in Section 8.2.2.5.

Prepare sulfide working standard A by diluting 1.00 ml of sulfide stock standard to 1000 ml. This solution contains 1.6 mg sulfide/L, if the concentration of the sulfide stock standard is exactly 1600 mg/L.

8.7. AVS Evolution reagents

8.7.1. Hydrochloric acid 6M - Dilute 500 ml of concentrated hydrochloric acid to 1.0 L de-aerated reagent water.

8.7.2. Nitrogen gas, oxygen free, with regulator and flow controller.

8.7.3. Plastic hypodermic syringe, 30 ml, and needle.

8.8. Colorimetric reagents

8.8.1. Sodium Hydroxide solution, 0.5N - Dissolve 20 g sodium hydroxide per 1000 ml reagent water. Generally make 4.0 L at one time. (80 g NaOH to 4.0 L)

8.8.2. Mixed diamine reagent, MDR

8.8.2.1. Component A - Add 1320 ml concentrated sulfuric acid to 680 ml of reagent water. N-N-dimethyl-p-phenylenediamine oxalate in it.

8.8.2.2. Component B - Dissolve 10.8 g ferric chloride hexahydrate ($FeCl_3 \cdot 6\,H_2O$) in 200 ml concentrated hydrochloric acid and dilute to 400 ml with reagent water.

8.8.2.3. Mix components A and B.

8.9. Sulfuric acid solution, 1.0M - Dilute

9. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10. PREVENTATIVE MAINTENANCE

10.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

10.2. Wipe down the Midi unit after use to remove any residual acid.

11. PROCEDURE

11.1. Macro distillation procedure

11.1.1. Place two neck flasks on stir plates and clamp into place.

11.1.2. Add one stir bar and 100 ml of reagent water to each flask. The amount of water added and the water present in the wet sediment should not exceed 120 mls.

11.1.3. Add 80 mls of 0.5N NaOH to each of the two towers for each flask. Label the first tower A, the second tower B.

11.1.4. Place septum stopper and impinger in two-neck flask. Place tower scrubbers in towers and connect the glassware with the tubing.

11.1.5. Purge the system with N_2 gas for approximately 10 minutes.

11.1.6. Weigh 10 g of wet sediment onto a 2x2 inch piece of parafilm. Remove the septum stopper and place the parafilm with sample into the flask. Do not rinse sample into flask. Purge the system for another 10 minutes.

Stop the flow of gas. Using the 30 ml hypodermic syringe, add 20 ml of 6M HCl to the flask via septum. Bubble nitrogen through the system for one hour while constantly stirring. For the LCS and spike, add the appropriate amount of sulfide standard before adding the acid.

11.1.7. Calibration and Standardization

11.1.7.1. Prepare a sulfide curve as follows:

11.1.7.1.1. Add 80 mls of 0.5N NaOH to each of 6 100 ml class volumetric.

11.1.7.1.2. Using working standard A, add 1 ml, 2 ml, 3 ml, 4 ml and 5 ml to each flask. Do not add sulfide standard to the first flask.

11.1.7.1.3. Calculate concentration of standards as follows:

$$\frac{(mls\ STD\ added) \times (Conc.\ of\ STD)}{Final\ volume\ (100ml)} = \_\_\ mg/L$$

Example: $\frac{4\ ml \times 16\ mg/L}{100\ ml} = 0.64\ mg/L$

11.1.7.1.4. Use the 0 ml STD for the CCB and the 4 ml STD for the CCV.

11.1.8. After one hour has elapsed, remove the scrubber from tower B and transfer to a 250 ml plastic container. Repeat for tower A. Leave N_2 gas flowing through scrubbers so that the NaOH does not go back up into the scrubber.

11.1.9. Disconnect tubing from flow meter at the flask impinger and then turn off the N_2 at the flow meter. This prevents liquid from backing up into the flow meter and destroying it.

11.2. Midi distillation procedure

11.2.1. Rinse scrubbers with 6N HCl follwoed by a rinse with deionized water.

11.2.2. Place boiling tubes, with impinger and septa inserted, into the midi system. Connect gas lines to impingers. Clamp septum to impinger.

11.2.3. Add 40mL of 0.5N NaOH to each bubbler vessel and clamp scrubber to bubbler vessel. Connect scrubber, with bubbler vessel attached, to the impinger arm.

11.2.4. Purge the system for approximately 10 minutes.

11.2.5. Add 10.0g of sample (or less depending on expected sulfide concentration) to the boiling tube. Rinse sample to the bottom of the boiling tube with 40mL of reagent water. Make sure no sample remains above the reagent water level. Clamp the impinger to the boiling tube.

11.2.6. Perform calibration steps as described in section 11.1.7.

11.2.7. Purge system for 10 minutes.

11.2.8. Connect scrubber tube with a disposable pipet to scrubber extension. Place the pipet into 50mL centrifuge tube filled with 40mL of 0.5N NaOH. (These are the "B" towers.)

11.2.9. Purge for 10 minutes.

11.2.10. Add 10.0 mL of 6N HCl to each boiling tube through the septum using a 10 mL syringe.

11.2.11. Let system react for 1 hour.

11.2.12. Place scrubber solution from bubbler vessels into separate containers. Before screwing on the cap, cover each solution with nitrogen.

Note: The same procedure is used for B towers except that the scrubber solution is left in the original centrifuge tube.

11.2.13. After complete, take down the apparatus and rinse all parts with water. Use a damp paper towel to wip down midi still and surrounding area.

Note: If Simultaneously Extracted Metals are to be determined, swirl bubbler tube to homogenize sample and pour 50 mL into a centrifuge tube for metals analysis.

11.3. Color development and analysis

11.3.1. If the sample is known or suspected to fall within the range of the curve, transfer the entire volume of tower A to a 100 ml volumetric flask. Add 10 ml of MDR and dilute to 100 ml with reagent water. For the six standards for the curve, add 10 ml MDR and dilute to 100 ml with reagent water. These volumes listed are for the macro setup. For midi procedure, volumes are halved.

11.3.2. If the samples are known or are suspected to be high in concentration, dilutions may be performed as follows.

Calculate the dilution based on a 50 ml volume. Subtract the amount of scrubber solution to be analyzed from 40 mls. Add that amount of 0.5N NaOH to a 50 ml volumetric. Add the volume of scrubber solution to be analyzed. Add 5 mls of MDR and dilute to 50 mls with reagent water.

Example dilution:

2 ml → 50 ml dilution

Place 38 mls of 0.5N NaOH in a 50 ml volumetric. Next place 2 ml of solution form tower A for that sample into the same volumetric. The volume in the flask is now 40 mls. Add 5 mls MDR and dilute to 50 mls with reagent water.

11.3.3. If blue color change develops in the sample, the scrubber solution of tower B for that sample must also be analyzed. If no color change occurs for tower A, then tower B does not have to be analyzed.

11.3.4. For tower B, place the entire 80 mls of scrubber solution in a 100 ml volumetric flask, add 10 mls MDR and dilute to 100 mls with reagent water.

11.3.5. After adding the MDR, allow a full thirty minutes for color development, but read the samples before 2 hours have elapsed.

11.3.6. Allow a thirty minute warm-up of the spectrophotometer. Zero the instrument with D.I. water at 670.0 nm.

11.3.7. Calibrate the instrument with the prepared curve starting with the low standard. A calibration with a correlation coefficient (r) of ≥ 0.995 must be obtained. Corrective action for a correlation coefficient < 0.095 is to discard existing curve, obtain new volumetrics and make a new curve. Verify concentration of stock sulfide standard.

11.3.8. Analyze samples in the following order:

Analysis	Sample	Analysis	Sample
1	LCS-1	15	CCV-2
2	LCS-2	16	CCB-2
3	CCV-1	17	Sample-7
4	CCB-1	18	Sample-8
5	MB-1	19	Sample-9
6	Sample-1	20	Sample-10
7	Sample-1D	21	Sample-11
8	Sample-1S	22	Sample-12
9	Sample-1SD	23	Sample-13
10	Sample-2	24	Sample-14
11	Sample-3	25	Sample-15
12	Sample-4	26	Sample-16
13	Sample-5	27	CCV-3
14	Sample-6	28	CCB-3

11.3.9. Analyze a continuing calibration verification standard (CCV) and continuing calibration blank (CCB) every 10 sample or QC analyses. The CCV is the fifth point on the curve. The CCV must be $\pm$ 10% of the expected value. If the CCV fails, then the curve is not

correct. The correlation coefficient may be ≥ 0.995, but if the slope is to high or too low, then the CCV will not pass. Notify supervisor, re-make the curve and re-calibrate. The CCB must be < 0.01 ppm - The CCB is the first point of the curve.

11.3.10. Calculate AVS concentration as follows:

$$\frac{(Conc.\ from\ curve)\ (DF)\ x\ Final\ volume}{Initial\ wt.}\ x\ \%solids\ =\ ppm\ AVS$$

11.3.11. Final volume will always be 100 mls, unless a volume less than 80 mls is used in the tower scrubbers.

12. QA/QC REQUIREMENTS

12.1. Refer to the SOP for Analytical Batches and Analytical Sequences for guidelines for frequency of QC sample analyses. The QC samples required are as follows:

12.1.1. A method blank (MB) for every 20 samples analyzed.

12.1.2. One LCS for every 20 samples analyzed. Due to the time requirements for set-up and distillation, it is best to distill 2 LCS's for every 20 samples. Sulfides are very unstable and easily degraded and therefore it is not uncommon to have low sample recoveries.

12.1.3. Analyze one matrix spike and one matrix spike duplicate per 20 samples minimum. See 10.1.2 for explanation.

12.1.4. Analyze one sample duplicate for every 20 samples minimum.

12.2. Acceptance Criteria

12.2.1. The method blank (MB) must be < MRL. If MB > MRL, reanalyze MB. Try to determine source of contamination.

12.2.2. MRL calculated as follows:

$$\frac{0.05\ mg/L\ x\ Final\ volume(L)}{Initial\ wt.(kg)}\ x\ \%solids$$

12.2.3. The LCS must be ± 15% of the expected value. If the LCS > ± 15%, sulfides were lost at some point during distillation, or sample was spiked at the improper level. Re-analyze

LCS and all samples associated with that LCS. Notify supervisor. (Note - LCS's generally only fail high if the sample was spiked too high).

12.2.4. Matrix Spike recovery criteria is 75-125% recovery. If recovery is outside the criteria, re-analyze spike. If two spikes were analyzed initially, then matrix interferences caused low spike recoveries. No need to reanalyze at that point. Notify supervisor.

12.2.5. The relative percent difference (RPD) for duplicates should be $\leq$ 20% RPD. If the RPD is > 20%, verify homogeneity of sample. Reanalyze the duplicate. Notify supervisor. Calculate RPD as follows:

$$RPD = \frac{High\ value\ -\ Low\ value}{Average} \times 100$$

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Calculate samples results as described in section 11.6.12. Results for QC analyses are calculated and recorded as specified in section 12. Average, RPD, spike level and spike recovery are entered on benchsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.2. The appropriate benchsheets, located in Appendix A, should be in use at all times during AVS analysis.

13.3. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.4. Refer to the SOP for Laboratory Data Review Process for general guidelines for data review.

13.5. Reporting

13.5.1. Soils, sediments, and other solid matrices are reported as mg/Kg, either dry weight or as received, according to the project requirements.

13.5.2. The MRL is calculated as follows:

$$\frac{0.05\ mg/L \times Final\ volume(L)}{Initial\ wt.(kg).} \times \%solids$$

13.5.3. The MRL will vary depending on % solids content. For samples reported as received, the MRL = 0.05 mg/Kg.

13.5.4. For samples that did not require a dilution, 3 significant figures will be reported. For samples that required a dilution, report only 2 significant figures.

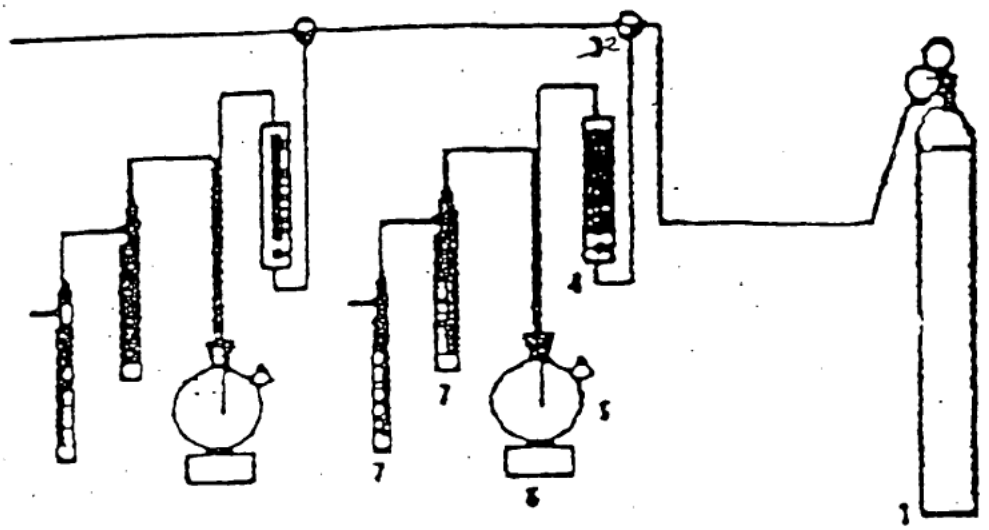
13.5.5. For samples below the MRL, report the number as < MRL.

14. REFERENCES

Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, August, 1991

FIGURE 1

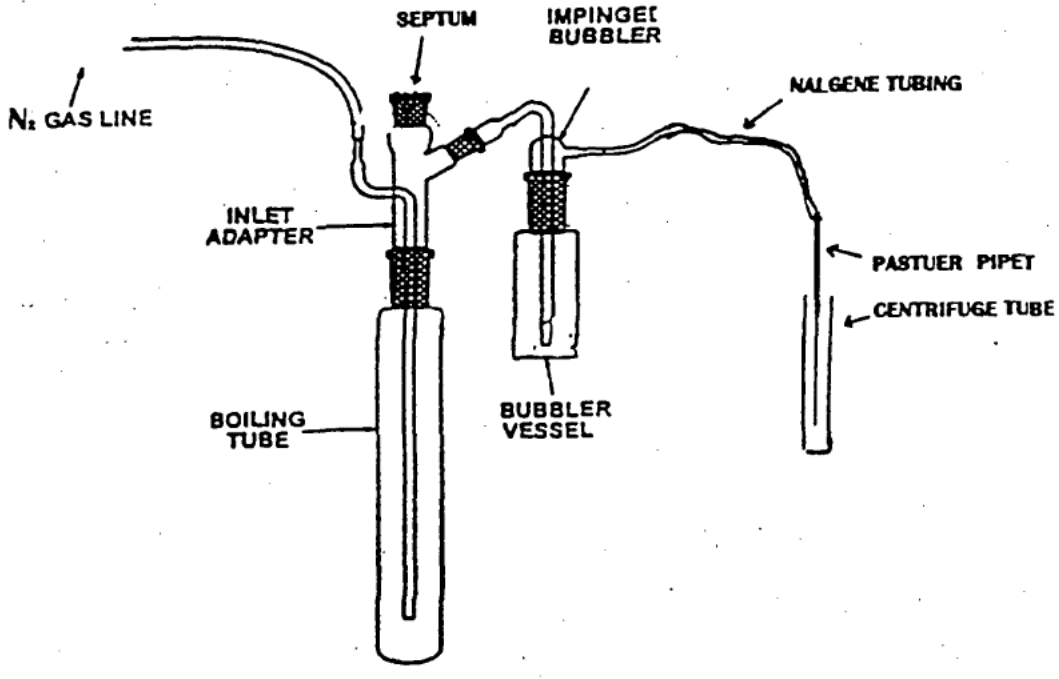
Macro Distillation Apparatus



Apparatus for AVS determination: 1 N_2 cylinder, 4. Flow controller, 5. Reaction flask, 6. Naguefic stirrer, 7. Impingers with non-fritted outlets.

FIGURE 2

Midi Distillation Apparatus



APPENDIX A

Benchsheet

COLUMBIA ANALYTICAL SERVICES, INC.

9030M/EPA Draft Aug. 1

Work Order #.: _____ Method: Methylene Blue Colorimet

Analysis: Total Sulfides/ AVS/ Soluble Sulfides

Date Prepared	Sample Name / Lab Code	Initial Wt./Vol (g) or (ml)	inal Volum (ml)	mg/L (in solution)	mg/L - mg/kg As Rec'd	% Solids	mg/kg Dry Wt.

LCS 1 = % REC =

LCS 2 = % REC =

Spike = % REC =

Spike Dup. = % REC =

x =

STD ID# = Conc. = RPD =

Prepared By:	Date Prepared:
Analyzed By:	Date Analyzed:
Reviewed By:	Date Reviewed:

COLUMBIA ANALYTICAL SERVICES, INC.

EPA Draft, Aug. 1991

Work Order #.: _____ Method: Methylene Blue Colorimetric

Analysis: AVS/SEM

Date Prepared	Sample Name / Lab Code	Initial Wt./Vol (g) or (ml)	Final Volume (ml)	mLs 50% HCl added	%HCl in Solution	% Solids	mg/kg Dry Wt.

LCS 1 = % REC =

LCS 2 = % REC =

Spike = % REC =

Spike Dup. = % REC =

x =

STD ID# = RPD =

Prepared By:	Date Prepared:
Analyzes By:	Date Analyzed:
Reviewed By:	Date Reviewed:

STANDARD OPERATING PROCEDURE

TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION

GEN-9030M

Revision 3

May 2, 1998

Approved By:

Supervisor	5/12/98 Date
QA Coordinator	5-12-98 Date
Laboratory Manager	5-22-98 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
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Standard Operating Procedure

for

TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION

1 SCOPE AND APPLICATION

1.1 The distillation procedure described in this method is designed for the determination of sulfides in aqueous, solid waste materials, or effluents. This method provides only a semi-quantitative determination of sulfide compounds considered "acid-insoluble" (e.g., CuS and SnS_2) in solid samples. Recovery has been shown to be 20 to 40% for CuS, one of the most stable and insoluble compounds, and 40 to 60% for SnS_2 which is slightly more soluble.

1.2 This method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure which can be analyzed by Method 9031.

1.3 Method 9030M is suitable for measuring sulfide concentrations in samples which contain between 0.2 and 50 mg/kg of sulfide. The Method Reporting Limit (MRL) for water is 2 mg/L and 4 mg/kg for soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) in water has been determined at 0.05 mg/L. The soil MDL (soluble sulfide) has been determined at 3 mg/kg.

1.4 This method is not applicable for distilling reactive sulfide, however, Method 9030M is used to quantify the concentration of sulfide from the reactivity test. Procedures for titration quantification by 9030A are included.

1.5 This method measures total sulfide which is usually defined as the acid-soluble fraction of a waste. If, however, one has previous knowledge of the waste and is concerned about both soluble sulfides such as H_2S, and metal sulfides, such as CuS and SnS_2, then total sulfide is defined as the combination of both acid-soluble and acid-insoluble fractions. For wastes where only metal sulfides are suspected to be present, only the acid-insoluble fraction needs to be performed.

2 SUMMARY OF METHOD

2.1 For acid soluble sulfide samples, separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample. The sample is heated to 70°C and the hydrogen sulfide (H_2S), which is formed, is distilled under acidic conditions and carried by a nitrogen stream into 0.5N NaOH.

2.2 The trapped sulfides are reacted with N-N-dimethy-p-phenylenediamine to form methylene blue, which is then read at 670nm.

3 DEFINITIONS

Laboratory Control Sample (LCS) - a solution of prepared in the laboratory which goes through all steps of the analysis that a sample does, and is used to determine if the analysis is in control.

Method Blank (MB) - a solution of the laboratory prepared deionized water that is carried through analysis like a sample, to serve as a measure of contamination associated with laboratory storage, preparation, or instrumentation.

Continuing calibration verification standard (CCV) - a solution of prepared in the laboratory at approximately the midpoint of calibration curves. CCV's are analyzed to verify that the instrument performance has not changed during the course of the analytical run.

Continuing calibration blank (CCB) - a blank solution of deionized water. CCB's are analyzed to verify that the instrument has not become contaminated during the course of the analytical run.

Sample Duplicate - a second aliquot of a sample that are treated exactly the same throughout laboratory analytical procedures. The purpose is to verify the precision associated with the laboratory procedures. The Relative Percent Difference (RPD) should not exceed 20%.

Matrix Spike - aliquots of sample to which known amounts of an analyte of interest has been added. These are treated exactly the same throughout laboratory analytical procedures. The purpose of a matrix spike is to determine whether the sample matrix contributes bias to the analytical results.

Analytical Run Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with the instrument calibration or calibration verification followed by samples interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Refer to the SOP for Analytical Batches and Analytical Sequences for description of applicable batching procedures.

4 INTERFERENCES

Contact with oxygen must be avoided in all stages from sampling to analysis.

5 SAFETY

5.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard, and exposure should be minimized.

5.2 Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or well ventilated area. If a high concentration of hydrogen sulfide is detected in the air by the laboratory staff, sample handling procedures must be corrected. According to Sax (9), an air concentration of 10 ppm of H_2S is permitted for an 8 hour shift for 40 hours per week.

5.3 If samples originate from a highly contaminated area, appropriate sample handling procedures to minimize worker exposure must be followed.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addressed the consideration discussed in Chapter 9 of EPA Reference SW846.

6.2 All aqueous samples and effluents must be preserved with zinc acetate and sodium hydroxide. Use four drops of 2N zinc acetate solution per 100 ml of sample. Adjust the pH to greater than 9 with 6N sodium hydroxide solution. Fill the sample bottle completely and stopper with a minimum of aeration. The treated sample is relatively stable and can be held for up to seven days. If high concentrations of sulfide are expected to be in the sample, continue adding zinc acetate until all the sulfide has precipitated. For solid samples, fill the surface of the solid with 2N zinc acetate until moistened. Samples must be cooled to 4°C and stored headspace free.

6.3 Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

6.4 During storage, sulfide can be formed or lost due to biological activity, and sulfide can be lost by volatilization or oxidation.

6.5 Samples should be cooled to 4°C as soon as possible after collection. Samples maintained at have been found to have no significant loss for storage periods up to 2 weeks. Holding time samples should not exceed 14 days for soil and other solid matrices, and 7 days for waters.

7 APPARATUS AND EQUIPMENT

7.1 Gas Evolution Apparatus.

7.2 Two sulfide towers and sulfide tower scrubbers

7.3 3-neck flask, 500 ml

7.4 Purge gas outlet/inlet/sample impinger

7.5 Addition flask

7.6 Tubing - ¼ in o.d. Teflon or polypropylene. Do not use rubber.

7.7 Hot plate stirrer

7.8 Quart Pyrex bowl

7.9 Nitrogen regulator

7.10 Flow meter

7.11 Top loading balance - capable of weighing 0.1g.

7.12 Spectrophotometer capable of reading a wavelength at 670nm.

8 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 Reagent Water - All water used in this method must be free of dissolved oxygen and sulfide. Prepare reagents and oxygen free water daily. Purge D.I. water for approximately 1 hour with nitrogen gas.

8.2 Sodium Sulfide Standard

8.3 Sulfide stock standard solution, approximately 0.05 m or 1600 mg/L.

Weigh about 12 grams of Na_2S-$9H_2O$ and dissolve it in 1,000 ml of reagent water. Store in a brown bottle. To prevent air oxidation, the sulfide solution should be maintained under oxygen-free nitrogen or argon.

8.4 Standardize against thiosulfate solution.

8.4.1 Pipette 10.00 ml of 0.025N standard iodine solution into each of two 125-ml Erlenmeyer flasks.

8.4.2 Pipette 2.00 ml of sulfide stock standard solution into one flask. Pipette 2.00 ml of deionized water, as a laboratory reagent blank, into the other flask.

8.4.3 Add 5.00 ml of 6M HCl into each flask, swirl slightly, then cover and place in the dark for 5 minutes.

8.4.4 Titrate each with 0.025N thiosulfate (Section 7.7) until the yellow iodine color fades to a pale straw. Just before all the iodine has been titrated, add starch indicator (Section 7.6) dropwise to form a pale blue color. Continue the titration with the thiosulfate. The end point is reached when the blue color first disappears.

8.4.5 Calculate the sulfide concentration as follows:

$$Sulfide\ (\mu g / mL) = \frac{(T_{blank} - T_{sample}) \times N}{V_{sample}} \times \frac{1\ mole\ S^{2}}{2\ equiv\ S^{2}} \times \frac{1000\ \mu moles}{1\ mmole} \times \frac{32 \mu g}{\mu mole}$$

where T = volume of titrant used for the blank and sample (ml)
N = Normality of S_2O_3 titrant
V = volume of sample used (ml), (2.00 ml) recommended

8.5 Iodine Solution (Approximately 0.025N)

Dissolve 25g potassium iodine, KI, in 700ml of reagent water. Add 3.2g iodine, I_2. Allow to dissolve. Add 2ml of 6N HCL. Dilute to 1.0L with reagent water.

8.6 Starch Solution - Dissolve 1g soluble starch powder in 100 mls of hot reagent water.

8.7 Sodium Thiosulfate Solution (0.025N)

Purchase as a standardized solution. Alternatively, dissolve 6.205 ± 0.005g $Na_2S_2O_3 \cdot 5H_2O$ in 500 ml reagent water. Add 18ml 0.5N NaOH and dilute to 1 liter with reagent water and standardize the solution.

8.8 0.5M NaOH - Place 3 liters of reagent water in cubitainer. Dissolve 80.0g NaOH in the water. Add 1 liter of reagent water and mix well.

8.9 Concentrated Sulfuric Acid (H_2SO_4 36N)

8.10 Mixed Diamine Reagent - MDR

8.10.1 Component A - Add 1320 ml concentrated sulfuric acid to 680ml of reagent water. After solution cools, dissolve 4.50g N-N-dimethy-p-phenylenediamine oxalate in it.

8.10.2 Component B - Dissolve 10.8g ferric chloride hexahydrate ($FeCl_3$4,3·6 H2O) in 200 ml concentrated hydrochloric acid and dilute to 400 ml final volume with reagent water.

8.10.3 Mixed diamine reagent, MDR - Mix components A and B.

9 RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10 PREVENTATIVE MAINTENANCE

10.1 No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

10.2 Follow the manufactuer's procedure for spectrophotometer maintenance.

11 PROCEDURE

11.1 Distillation

11.1.1 Apparatus Preparation - Set up glassware as shown in figure 1.

11.1.2 After glassware is set-up, add enough water to cover the impinger. Turn on N_2 and purge system for 10-15 minutes. If analyzing water by this method, purge the system without water in it. Turn on hot plate and heat water in pyrex bowl to 70°C.

11.1.3 Add 10g of sample to flask. Check stir rate to make sure sample is stirring. Re-close flask and check bubble rate in towers. Allow system to purge for 5 minutes.

11.1.4 Add 100mls of water sample to flask. If necessary, add additional reagent water to ensure impinger is under the water. Close flask and purge system for 5 minutes.

11.1.5 Add standard solutions to LCS and spike(s).

11.1.6 Remove pinch clamp on N_2 tubing to addition funnel. Open stopcock and add sulfuric acid drop-wise at a rate of about 2-3 drops per second.

11.1.7 After all the acid is in the sample flask, close the stopcock and close-off N_2 to the addition flask.

11.1.8 Allow samples to distill for 90 minutes.

11.1.9 After 90 minutes, remove the scrubber from tower B, then remove the scrubber from tower A. Remove the tubing from the impinger, then turn off N_2 to that apparatus.

11.2 Quantification - Method 9030A, 9030M

11.2.1 Spectrophotometric determination - Method 9030M

11.2.1.1 Transfer the 80.0 mls of scrubber solution from tower A to a class A 10 volumetric flask, or transfer an aliquot and dilute to 80.0 ml in 0.5N NaOH. If samples are expected to be high, or known to be of a high concentration, all volumes may be divided in half.

11.2.1.2 Add 10mls MDR to the scrubber solution, and reagent water to 100 mls. Stopper flask and shake to mix well. If using only 40mls of scrubber solution, add only 5mls MDR to solution and reagent water to 50mls.

11.2.1.3 Allow 30 minutes for full color development, but read samples before 2 hours have expired after adding MDR.

11.2.1.4 Calibration and Standardization

11.2.1.4.1 Turn on spectrophotometer and allow to warm up at least 30 minutes prior to reading samples.

11.2.1.4.2 Determine the concentration of the sulfide stock solution as per 7.4. From this value, make a working standard by adding 1.0ml of stock standard to 99.0ml reagent water. This standard is unstable and should be prepared fresh daily.

11.2.1.4.3 From the working standard, create a 6 point curve as follows (see the tab below for an example): Add 80.0mls 0.5N NaOH to the 100.0ml class A

volumetric. Add the appropriate mls of working standard to the appropriate flask. Add 10.0mls MDR and reagent water to 100.0ml final volume. Allow color to develop for 30 minutes and read. Prior to analyzing the curve, zero the spectrophotometer at 670nm using D.I water. The 0.0ml curve standard may be used as the CCB and 4.0ml curve standard may be used as the CCV.

mls Working Standard		Conc. Working STD	Vol.	Conc. of Curve
CCB	0.0	A	100ml	0 mg/L
	1.0	A	100ml	0.20 mg/L
	2.0	A	100ml	0.40 mg/L
	3.0	A	100ml	0.60 mg/L
CCV	4.0	A	100ml	0.80 mg/L
	5.0	A	100ml	1.0 mg/L

A = concentration of working standard.
Example: Working standard = 20mg/L.
1.0ml x 20.0 mg/L/100ml = 0.20mg/L
3.0ml x 20.0 mg/L/100ml = 0.60mg/L

11.2.1.4.4A linear regression correlation coefficient of 0.9950 or better for the calibration curve is required.

11.2.1.4.5The CCV acceptance criteria is 100% ± 10% of the true value. If the CCV fails, the slope of the curve is probably too low and the curve should be reanalyzed

11.2.1.4.6Due to instability of sulfide stock standard, the concentration should be verified prior to creating the working standard. The value of the stock standard will decrease daily and should be made fresh before the value drops below about 1300mg/L.

11.2.1.5Calculations

11.2.1.5.1Enter the value of the absorbance into the curve and determine the sample concentration.

11.2.1.5.2Solution Conc. x Final Volume/Init. vol. or wt. = Conc. in sample

11.2.1.5.3Conc. in sample is final number for water matrix

11.2.1.5.4Conc. in sample/% Solids (decimal form) = Dry weight value

11.2.1.6The Method Reporting Limit (MRL) is calculated as follows:

Water:

$$\frac{0.05\ mg/L \times Final\ volume(L)}{Initial\ vol.(L)}$$

Soil:

$$\frac{0.05\ mg/L \times Final\ volume(L)}{Initial\ wt.(kg)} \times \%solids$$

11.2.2 Titration determination, Method 9030A

11.2.2.1Determine I_2 concentration by standardizing against 0.025 N sodium thiosulfate.

$$N(I_2) = \frac{ml\ of\ titrant \times N\ of\ titrant}{ml\ of\ I_2\ titrated}$$

11.2.2.2Pipet enough I_2 into 100 ml beaker (1 nd, 2 ml, 3 nd, etc).

11.2.2.3Add 10 ml 6 N HCl.

11.2.2.4Aliquot 25 ml distillate, using volumetric pipet, and dispense into beaker, below the surface of the I2 solution (if color is pale start over with more I_2).

11.2.2.5Put in a stir bar.

11.2.2.6Titrate until pale yellow.

11.2.2.7Add 2-3 drops starch until black.

11.2.2.8Continue titrating until colorless endpoint and take reading.

11.2.2.9Calculate amount of sulfide, using formula below:

$$Sulfide\ mg/Kg = \frac{[(ml\ of\ I_2 \times N\ of\ I_2) - (ml\ of\ titrant \times N\ of\ titrant)](16.03) \times Scrubber\ Vol.\ (m}{sample\ weight\ (Kg) \times ml\ of\ scrubber\ titrated}$$

12 QA/QC REQUIREMENTS

12.1 QC Samples Required

12.1.1 A method blank (MB) and laboratory control sample (LCS) should be analyzed with each batch (twenty samples).

12.1.2 A duplicate sample and one matrix spike should be analyzed with each batch. The same duplicate and matrix spike may be used over a 5 day period, as long as the twenty sample batch is not exceeded.

12.1.3 The LCS and matrix spike are generally spiked to achieve the same concentration as the CCV. (i.e. 4mls working standard in 100mls sample, or 10.0 grams soil sample). If the sample is known or suspected of being a high concentration, a second working standard is prepared. This working standard is 10 times higher in concentration than the first working standard and is prepared by placing 10.0mls stock sulfide standard solution in 90.0mls reagent water.

12.2 Acceptance Criteria

12.2.1 Sample duplicates: <20% RPD

12.2.2 Sample matrix spike recovery: 55-125%

12.2.3 Method blank should be <MRL

12.2.4 LCS - 100% ± 30%

12.3 Corrective Action Requirements

12.3.1 LCS - Generally two LCSs are distilled per batch due to instability of sulfides and difficulty in obtaining recoveries. In most cases, both will be within acceptance criteria. If not, redistill and analyze additional LCS. Check with supervisor.

12.3.2 Matrix Spikes - Generally, two matrix spikes are analyzed. If both matrix spikes fail, matrix interference has been verified. If one MS fails and one MS passes, a third MS should be analyzed. Check with supervisor.

12.3.3 Replicate samples: run sample in triplicate.

13 DATA REDUCTION, REVIEW, AND REPORTING

13.1 Reporting Units

13.1.1 Water samples are reported as mg/L sulfide.

13.1.2 Soil samples are reported as mg/Kg dry weight sulfide.

13.2 Method Reporting Limits (MRLs)

The limit of detection is 0.01 on the spectrophotometer.

Water - 0.05 x 100ml/100ml = 0.05 mg/L
Soil - 0.05 x 100ml/10.0g/lowest % solids

(see section 11.2.1)

13.3 Significant Figures and Reporting Values Below Reporting Limits

Report 3 significant figures unless a dilution is performed, then report 2 significant figures.

13.4 If the calculated sample concentration is below the MRL, report the value as less than the calculated MRL. Not detected (ND) will go on the report form.

14 REFERENCES

14.1 Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, December 1991.

14.2 EPA Method 9030A Acid-Soluble and Acid-Insoluble Sulfides, EPA SW-846 Revision 1, July 1992.

FIGURE 1

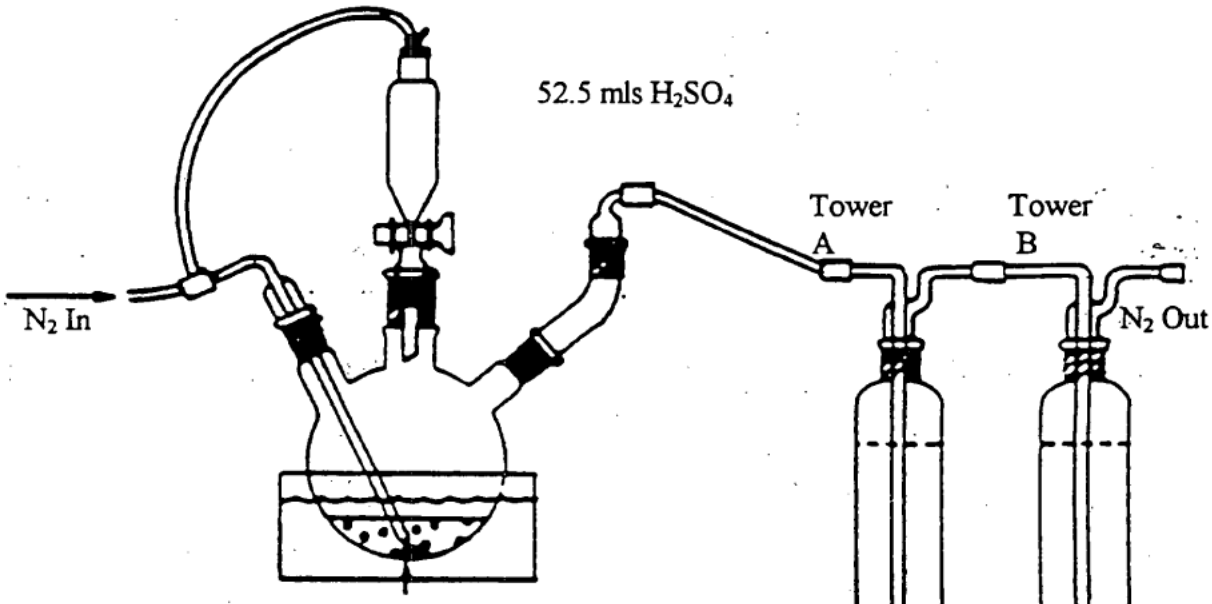
52.5 mls H_2SO_4
Tower A
Tower B
N_2 In
N_2 Out
Hot Water Bath
with Magnetic Stirrer
Stirring Bar
80.0 mls 0.5N
NaOH Scrubbing
Bottles

TABLE I

Typical Run Scheme

Step	Sample
1	LCS
2	CCV-1
3	CCB-1
4	MB
5	Spl-1
6	Spl-1Dup
7	Spl-1MS
8	Spl-2
9	Spl-3
10	Spl-4
11	Spl-5
12	Spl-6
13	Spl-7
14	CCV-2
15	CCB-3
16	Spl-8
17	Spl-9
18	Spl-10
19	Spl-11
20	Spl-12
21	Spl-13
22	Spl-14
23	Spl-15
24	Spl-16
25	Spl-17
26	CCV-3
27	CCB-3

Repeat steps 5-27 for remaining samples

APPENDIX A

Benchsheets

COLUMBIA ANALYTICAL SERVICES, INC.

Work Order #.: _____

Method: 9030M/EPA Draft Aug. 19__ Methylene Blue Colorin

Analysis: Total Sulfides/ AVS/ Soluble Sulfides

Date Prepared	Sample Name / Lab Code	Initial Wt./Vol. (g) or (ml)	Final Volume (ml)	mg/L (in solution)	mg/L - mg/kg As Rec'd	% Solids	mg/kg Dry Wt.

LCS 1 = % REC =

LCS 2 = % REC =

Spike = % REC =

Spike Dup. = % REC =

$\bar{x}$ =

STD ID# = RPD =

Prepared By:	Date Prepared:
Analyzes By:	Date Analyzed:
Reviewed By:	Date Reviewed:

COLUMBIA ANALYTICAL SERVICES, INC.

Work Order #_________________________________

Method: 9030M
Methylene Blue Colorimetric
EPA DRAFT, Aug. 1991

Analysis for Total Acid Volatile/Dissolved Sulfide

LOW-RANGE CALIBRATION 0.0_________ ppmS^{2-}

Standard Conc. ppmS^{2-}							r =
Absorbance @ 670 nm.							S =
							I =

Lab Code							
Dilution (mL)							
Absorbance at 670 nm.							
ppm S^{2-} from Curve							
Total ppm S^{2-}							

Lab Code							
Dilution (mL)							
Absorbance at 670 nm.							
ppm S^{2-} from Curve							
Total ppm S^{2-}							

Lab Code							
Dilution (mL)							
Absorbance at 670 nm.							
ppm S^{2-} from Curve							
Total ppm S^{2-}							

Comments: Absorbances of all second scrubber solutions were below the MDL, for those samples in which the first scrubber was above the MDL. See strip charts for verification.

CCV = __________________ ppmS^{2-} % REC = __________________

Distilled by	Date Distilled
Analyzed by	Date Analyzed
Reviewed by	Date Reviewed

STANDARD OPERATING PROCEDURE

VOLATILE ORGANIC COMPOUNDS BY GC/MS

VOC-8260B
Revision 2
July 7, 1999

Approved By:

Supervisor	7-7-99 Date
QA Manager	7-7-99 Date
Laboratory Manager	7/7/99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
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Initials: _____ Date: _____
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Standard Operating Procedure
for

VOLATILE ORGANIC COMPOUNDS BY GC/MS

1. SCOPE AND APPLICATION

This procedure is used to determine the concentration of volatile organic compounds in water and soil using USEPA Method 8260B. This method may also be applicable to various types of aqueous and nonaqueous waste samples. Table 1 lists the compounds that can be determined by this method and the achievable method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) will vary depending on the instrument used.

2. METHOD SUMMARY

2.1. Discussion:

2.1.1. This procedure gives gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of parts per billion (ppb) levels of volatile organic compounds. A sample aliquot is injected into the gas chromatograph (GC) by either the purge and trap method or by direct injection. The compounds are separated on a wide bore fused silica capillary GC column. The compounds are detected by a mass selective detector (MSD), which gives both qualitative as well as quantitative information.

2.1.2. In the purge and trap process an inert gas, helium, is bubbled through the sample aliquot, at room temperature. This gas stream sweeps the volatile organic compounds out of the aqueous phase and into the gas stream - it purges the compounds out of the sample. The gas stream then passes through a sorbent column which selectively adsorbs, (traps) these compounds out of the helium. The preparation of soil samples (section 11.1.2) uses procedures described in USEPA Method 5030B or 5035. After the purging sequence is done, the sorbent column (the trap) is heated and backflushed onto the GC column. The GC column separates the compounds and passes then onto the MSD for identification and quantification.

2.1.3. The sensitively of this method depends on the level of background contamination (i.e. interferences) rather than on instrumental limitations. Highly contaminated waste samples will require a methanol extraction prior to analysis. This will elevate the reporting levels and may mask low levels of compounds of interest.

2.2. Deviations from the reference method(s): For water samples, a purge volume of 10mL is used, whereas the method (section 7.5.5) states 5 mL or 25 mL. The use of a 10 mL volume ensures sensitivity for "5 mL" type analyses *and*, on the analytical systems in use, meets the sensitivity goals of a 25 mL purge volume analysis. Also, the use of 10 mL rather than 25 mL decreases the negative effects of water being introduced into the P/T-GC-MS system.

3. DEFINITIONS

Analysis Window - Samples are analyzed in a set referred to as "a window". The window begins with the injection of the tune verification standard. After this standard has passed the method specific criteria a 12 hour analysis window is started. Next, a calibration curve or a continuing calibration standard (CCV see below) is run followed by a method blank. If both pass their specific criteria, then samples are run until the 12 hour time limit closes. A new window must then be opened and the sequence repeated.

Internal Standards - Internal standards are organic compounds which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to help calibrate the instrument's response and to compensate for slight purge flow fluctuations caused by running different purge vessel positions.

Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to the analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Method Blank (MB) - The method blank (also called continuing calibration blank) is a volume of clean reagent water analyzed on each GC/MS used for sample analysis. The purpose of the blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Interferences by common laboratory extraction solvents, such as Methylene Chloride, Acetone, and Freon 113 can cause problems. The area where volatile organic analyses are performed should be free of these solvents.

4.2. Other interferences include but are not limited to impurities in the inert purge gas, dirty plumbing/purge vessels, cross contamination by highly contaminated samples to clean ones in transport and storage, and carry over from one analysis to subsequent ones.

5. SAFETY

The toxicity or carcinogenicity of each compound or reagent used with this method is not known. Each compound, mix of standards, internal standards and surrogates as well as the samples should be treated as a potential health hazard. Exposure to each should be reduced to the lowest level possible through the use of gloves and a hood. Reference files of Material Safety Data Sheets (MSDS) are available to all personnel. CAS also has a file of the current OSHA regulations regarding the safe handling of the compounds specified in this method.

6. SAMPLE CONTAINERS, COLLECTION, PRESERVATIONS, AND STORAGE

6.1. All sample containers for volatile organic analyses should be washed with soap and water, deionized water rinsed, and baked at 105°C ± 5°C for approximately 2 hours prior to use. Alternatively, one can buy precleaned sample containers from major lab equipment suppliers. All containers should be of glass or amber glass and equipped with a screw top cap and PFTE (teflon) lined septa.

6.2. Collect all samples in duplicate, triplicate when possible. Prepare the proper number of sample bottles/containers prior to the sampling event with preservatives to adjust the samples pH to <2 with 1:1 HCI.

6.3. Slowly fill sample bottles to just overflowing taking care not to flush out the preservative or to entrain air bubbles in the samples. Seal the bottles with PFTE lined septa toward the sample and invert to check for entrained air bubbles.

6.4. Experimental evidence has shown refrigeration at 4°C alone will not stop biological degradation of some aromatic volatile organics. Adjusting the pH of the replicate samples

to less than two (pH <2) with 1:1 HCl (@ 2-3 drops per 40 mLs) preserves samples for 14 days after collection. Residual chlorine can also degrade some organic compounds, generating trihalomethanes (THM's).

6.5. All samples must be stored at 4 ± 2°C and must be analyzed within 14 days of collection. Any free product samples to be tested do not have any set holding times but should be analyzed as soon as possible.

7. APPARATUS AND EQUIPMENT

7.1. Gas chromatograph/Mass Selective Detector Systems

7.1.1. Each GC/MS system is set up with a GC suitable for subambient cooling of the GC column, injection onto a capillary column, and a stainless steel jet separator at the column's detector end prior to the transfer line interfaced with the MSD. Each MSD is a HP5970, HP5971, HP5972, or HP5973 that is controlled by the HP-MSDOS Chemstation software.

7.1.2. An alternate option to the use of a jet separator is to use a split/splitless injector and interfacing the capillary column directly into the MSD.

7.2. Purge and Trap with Autosampler

Each volatile GC/MS analytical system uses a purge and trap to introduce the sample onto the GC column. A Tekmar LSC-2 or its equivalent is needed. Each purge and trap has an autosampler (A/S) attached to run multiple samples, one at a time, and run unattended for extended periods of time. A Tekmar ALS 2016 or its equivalent for extended unattended automated running is needed.

7.3. GC Columns

Column 1: J&W Scientific DB-624 (or equivalent)
meters x 0.53 mm and fused silica column 1.5µm film thickness.

Column 2: Restex RTX-Volatiles (or equivalent) 60 M x 0.53mm id
fused silica column 2.0µm film thickness

Column 3: Restex RTX-Volatiles (or equivalent) 60 M x 0.32mm id
fused silica column 1.8µm film thickness

Column 4: Restex RTX-Volatiles (or equivalent) 30 M x 0.25mm id
fused silica column 1.4µm film thickness

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Methanol, purge and trap grade or equivalent.

8.2. Reagent water, prepared from deionized water, by charcoal filtration and then purging for approximately 2 hours prior to use.

8.3. Stock Standard Solutions

Commercially prepared and certified stock standards are used routinely for all the method specified analytes. All such mixtures are also routinely checked against an independent source for both analyte identification and analyte concentration. All such stock standard mixtures have expiration dates given by the manufacturer and must be replaced if the comparison with the independent check standards indicates a problem. Alternatively, stock standards may be prepared from neat chemicals. Store with minimal headspace, at -10° to -20°C and protect from light.

8.4. Working Standards - Prepare these standards from stock solutions. Prepare at concentrations which facilitate ease of preparation of instrumnet-level standards (calibration standards, etc.

8.5. Calibration Standards

8.5.1. A minimum of five different concentration levels for all the analytes are prepared by diluting working standards into reagent water. The lowest concentration level must be at the method reporting level, or a level corresponding to a sample concentration meeting project-specific data quality objectives, with the remaining four levels defining the working linear range of the analytical system. Refer to the front cover of the injection log for each instrument for detailed standard preparation instructions.

8.5.2. The suggested levels are 0.5, 2, 10, 20 and 40 ppb for waters; and 5, 20, 50, 100, and 200 ppb for soils. All calibration solutions are made up daily.

8.6. Internal Standards and Surrogates

The surrogates recommended are Dibromofluoromethane, toluene-d_8 and 4-bromofluorobenzene. The internal standards recommended are pentafluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene-d_4 and chlorobenzene-d_5. The other internal standards and surrogates may be used, depending on the analysis requirements. All surrogates and internal standards are added to every standard, sample, blank and spike at 10 ug/L for waters and 50 ug/L for soils.

8.7. Spiking Solutions

Matrix spike and laboratory control spike solutions must be prepared from a separate source than the calibration standards. Spiking solutions should contain the full list of analytes of interest. However, a subset may be reported.

Note: Refer to Table 2 for Standard Expiration Date Guidelines.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Purge and Trap /Autosamplers

9.3.1. The purge/trap system should be baked out and back-flushed daily as needed, generally prior to use on a daily basis.

9.3.2. Replace the trap monthly, or sooner if performance deteriorates.

9.4. Gas Chromatograph

9.4.1. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.4.2. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.5. Mass Spectrometer

9.5.1. Tune the MS as needed to result in consistent and acceptable performance (see section 11).

9.5.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.5.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8260B, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water Samples

11.1.1.1. No preparation is generally required, other than dilution with reagent water to bring analytes into the upper half of the calibration range. Thus, a 10 mL sample volume is run straight from the sample vial. See USEPA Methods 8260B and 5030B for further discussion.

11.1.1.2. All water samples must be checked to have a pH of 2 (pH < 2).

11.1.2. The analysis of a soil sample are broken into three types, the 5035 type, low-level 5030A type, and the mid-level type.

11.1.2.1. For 5035 analyses, one of the sampling options given in method 5035 is to be used. Depending on the option used, follow the instructions given in the method. The use of EnCore samplers is the preferred sampling technique.

11.1.2.2. The low-level type is a direct heated purge of soil using method 5030A guidelines. This requires its own separate initial calibration. For soil, 1-5 grams is weighed out into the sample vial and 5 mL of reagent water is added. QC spikes and internal standards are then added, and the sample is purged at a temperature of 40°C° ±1°. Calibration standards, LCS, and method blanks require 5grams Ottawa sand as the matrix.

11.1.2.3. The mid-level type is an extraction method and is only done when needed. In general, a five gram wet weight of soil is extracted with 10 mls of purge-and-trap methanol in a scintillation vial. Quickly add purge-and-trap methanol

and the surrogate spike (to total 10 mls) to the soil after transferring the soil aliquot to the vial. Cap and vortex until the sample is thoroughly mixed. A 1:50 dilution of this extract in water is run against the water calibration. The extract weight and volume used are recorded on a bench sheet, along with the methanol lot number.

11.2. Calibration

11.2.1. BFB Tuning Criteria - Each volatile GC/MS analytical system set up to run 8260B must meet the criteria listed in Table 3 for a 50 ng injection of BFB. Alternate tuning criteria (from Method 524.2 or CLP OLM03.1) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for all ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria. The analysis time for BFB is used to define the start of the 12-hour window in which all analyses must be performed.

11.2.2. GC/MS Analytical System Initial Calibrations

11.2.2.1. Prior to conducting any sample analyses, a 5 point calibration must be run. Recommended calibration levels are 0.5, 2, 10, 20, and 40 ppb for waters, and 5, 20, 50, 100, and 200 ppb for soils. Analyze each calibration standard and tabulate the area response of the characteristic quantitation ions versus concentration for each compound, internal standards and surrogate. Calculate the response factors (RF) for each compound and surrogate relative to the specified internal standard by:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

Where:

A_x = Area of the characteristic quantitation ion for compound x.

A_{ISTD} = Area of the characteristic quantitation ion for the specified internal standard.

C_x = The concentration of the compound added.

C_{ISTD} = The concentration of the specified internal standard.

11.2.2.2. Calculate the mean response factor $(\overline{RF_x})$ for each analyte and surrogate from the five calibration levels. Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$\%RSD = \frac{(SD)}{(\overline{RF_x})} 100.$$

11.2.2.3. The % RSD should be less than 15% for each compound. However, the % RSD for each individual CCC must be less than 30%. The CCC's are: 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, toluene, Ethylbenzene and Vinyl Chloride.

11.2.2.4. If a % RSD greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before reattempting calibration.

11.2.2.5. If the % RSD for any compound is 15% or less, linearity can be assumed over the calibration range, and the relative response factor for each analyte and surrogate is used to quantitate sample analytes.

11.2.2.6. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

- The mean of the RSD values for all analytes in the calibration standards is $\leq$ 15%. This is easily checked using the CALEXCEL command on the Enviroquant command line. After the Excel spreadsheet is displayed, use the function wizard to average the RSDs in a designated and labeled cell.

- The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.

- The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

11.2.2.7. If all of the conditions in Section 11.2.2.6 are met, then the average response factor may be used to determine sample concentrations as described in Section 13.1.

11.2.2.8. The response of 5 SPCC's must also be checked for their minimum $\overline{RF_x}$:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chrorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

11.2.2.9. After the 5 point calibration has passed all of the above criteria, and the mid-point (CCV) has been checked against the curve, then samples can be analyzed.

11.2.3. Following initial calibration, analyze an ICV standard if calibration standards have not been verified from a second source previously. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. The %D for all analytes must be ± 30%.

Some compounds may exceed this criteria and the initial calibration may still be valid. The analyst must use judgement when evaluating reactive compounds or those exhibiting poor chromatographic behavior. These may include the early gases, acrolein, and 2-chloroethyl vinyl ether. If a second source standard is not available or is cost prohibitive, then an independently prepared solution (prepared by analyst other than analyst preparing initial calibration standards) may be used as the ICV and must meet the criteria above.

11.2.4. Daily GC/MS Calibration

11.2.4.1. The start of a 12-hour analysis window requires a check of the MSD's tune via an injection of 50 ng of BFB. If the criteria found in Table 3 are met, then a check of the initial calibration curve is done. If the first run of the BFB fails, retry. If the second run also fails, inspect the system for potential maintenance needs. You may have to retune and recalibrate the system.

11.2.4.2. After the tuning criteria have been verified, the initial calibration must be checked and verified by analyzing a midrange calibration standard. The 10 ppb level for waters and 50 ppb level for soils is recommended. For 8260B, water daily check standards are 10µl of the 50 ppm 8260 working standard and ketone mix is spiked into 50 mL reagent water (section 8.2), and a 10 mL aliquot is purged. For 8260B soil daily check standards, 12.5 µl each of the 200 ppm DVM-580 standard and the 400/200 ppm ketone mix standard is spiked into 50 mL reagent water, and a 5 mL aliquot is purged. The results are compared with those of the initial calibration's $\overline{RF}$. The criteria for the SPCCs, as outlined above, must be met. Also, the CCCs must meet $\leq$ 20% drift from the initial calibration curve RFs.

11.2.4.3. If the tune criteria and the continuing calibration criteria are met, then the retention times of all compounds, surrogates, and internal standards are checked against the initial calibration. If the retention time for any internal standard

changes by more than 30 seconds from the retention time from the mid-point standard of the most recent initial calibration, the system must be inspected for malfunctions and corrections must be made, as required. If the area for any of the internal standards changes by a factor of 2 (-50% to +100%) from the retention time from the mid-point standard of the most recent initial calibration, corrections must be made to the system.

11.2.5. Quantitation of all compounds will be based on the initial calibration.

11.3. Identification of Analytes

The MSD data system software identifies a sample component by first finding and identifying the surrogate and internal standards. After they have been integrated, the extracted ion chromatogram is searched for all calibrated analytes. Any peak associated with the proper time window having the primary characteristic quantitation ion identified has it's results calculated. The analyst should follow interpretation guidelines in the method to make accurate identification of analytes. If there is no peak found for an analyte in the expected retention time window and the mass spectra does not match according to the method criteria, then the analyte is "not found". Print out spectra for all confirmed hits.

12. QA/QC REQUIREMENTS

12.1. 12-Hour Analysis Window Requirements

For every 12-hour analysis window, after meeting the tune and continuing calibration criteria, at least one method blank, one LCS, and one MS/DMS pair must be run for each matrix. Refer to the SOP for Analytical Batched and Analytical Sequences. Analytical windows must have at least one base (primary) sample analysis to require a MS/DMS pair.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples begins. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP for The Determination of Method Detecion Limits.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Acceptance Criteria

12.3.1. The acceptance criteria for tuning verification, initial, and continuing calibration verification have been outlined above in Section 11.

12.3.2. Appendix I contains the acceptance criteria for evaluating matrix spikes, LCS's and surrogate recoveries.

12.3.3. Corrective action requirements have been outlined above in Section 11. Also, the corrective action requirements of any project-specific project plan should be used when applicable.

13. DATA REDUCTION , REVIEW, AND REPORTING

13.1. Calculations

13.1.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. The results for a water sample are calculated as follows when $\overline{RF_x}$ is used:

$$A_x = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: A_x = the amount, in ppb, of the analytes in the sample;

$Resp_x$ = the peak area of the analytes of interest;

$Resp_{ISTD}$ = the peak area of the associated internal standard;

Amt_{ISTD} = the amount, in ppb, of internal standard added

$\overline{RF_x}$ = the average response from the five-point for the analytes of interest.

13.1.2. The results for low-level soil work are calculated by taking the normal print out, in ppb, (see the water results outlined above) and correcting for the total, dry soil sample actually purged:

$$(A_x) = \frac{(5\ grams)}{(ASW,\ gr)(\%\ Solids)} = A_x\ Low\text{-}Level\ Soil$$

Where: A_x = the amount, in ppb, from the data system; five grams is the nominal amount of soil that is heated and purged;

ASW_t = the actual soil wet weight, in grams, that is purged; and
% Solids the correction factor for dry weight.

13.1.3. Results for a high-level soil extract are calculated as follows:

$$(A_x) = \frac{(Dilution)(10\ ml)}{(ASW_t)(\%\ Solids)} = A_x\ High\text{-}Level\ Soil\ Amt.$$

Where: A_x = the data station results, in ppb;

Dilution = the dilution of the extract.

10 ml = the amount of methanol used to extract the soil;

ASW_t = the actual wet weight of soil extracted; and

% Solids = the dry soil correction.

13.2. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

13.3. Reporting

Reports are generated using Excel templates located in R:\VOA\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level. The detected analytes, surrogate and matrix spikes are then transferred, by hand, to the templates.

14. REFERENCES

14.1. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique*, U.S. EPA, SW-846, Final Update III, Method 8260B, Revision 2, December 1996.

14.2. *Purge and Trap*, U.S. EPA, SW-846, Final Updates I and III, Methods 5030A (Rev. 1) and 5030B (Rev. 2).

14.3. *Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste*, U.S. EPA, SW-846, Final Update III, Method 5035, Revision 0, December 1996.

TABLE 1

Method 8260B Analyte List

Compound	Water MRL	Soil MRL(low)	Soil MRL(mid)
	ug/L	ug/kg	mg/kg
1,1,1,2-Tetrachloroethane	0.5	5	.05
1,1,1-Trichloroethane (TCA)	0.5	5	.05
1,1,2-Trichloroethane	0.5	5	.05
1,1-Dichloroethane	0.5	5	.05
1,1-Dichloroethene	0.5	5	.05
1,1-Dichloropropene	0.5	5	.05
1,2-Dibromoethane (EDB)	2	20	0.1
1,2-Dichloroethane	0.5	5	.05
1,2-Dichloropropane	0.5	5	.05
1,3-Dichloropropane	0.5	5	.05
2,2-Dichloropropane	0.5	5	.05
2-Butanone (MEK)	20	20	1
2-Hexanone	20	20	1
4-Methyl-2-pentanone (MIBK)	20	20	1
Acetone	20	50	1
Benzene	0.5	5	.05
Bromochloromethane	0.5	5	.05
Bromodichloromethane	0.5	5	.05
Bromomethane	0.5	5	.05
Carbon Disulfide	0.5	5	.05
Carbon Tetrachloride	0.5	5	.05
Chlorobenzene	0.5	5	.05
Chloroethane	0.5	5	.05
Chloroform	0.5	5	.05
Chloromethane	0.5	5	.05
cis-1,2-Dichloroethene	0.5	5	.05
cis-1,3-Dichloropropene	0.5	5	.05
Dibromochloromethane	0.5	5	.05
Dibromomethane	0.5	5	.05
Dichlorodifluoromethane (CFC 12)	0.5	5	.05
Ethylbenzene	0.5	5	.05
Methylene Chloride	5	10	.25
Tetrachloroethene (PCE)	0.5	5	.05
Toluene	0.5	5	.05
trans-1,2-Dichloroethene	0.5	5	.05
trans-1,3-Dichloropropene	0.5	5	.05
Trichloroethene (TCE)	0.5	5	.05
Trichlorofluoromethane (CFC 11)	0.5	5	.05
Vinyl Chloride	0.5	5	.05

TABLE 1, continued

Compound	Water MRL ug/L	Soil MRL(low) ug/kg	Soil MRL(mid) mg/kg
Total Xylenes	1	5	0.1
Styrene	0.5	5	.05
Bromoform	0.5	5	.05
Isopropylbenzene	2	20	0.2
1,1,2,2-Tetrachloroethane	0.5	5	.05
1,2,3-Trichloropropane	0.5	5	.05
Bromobenzene	0.5	5	.05
n-Propylbenzene	2	20	.02
2-Chlorotoluene	2	20	.02
4-Chlorotoluene	2	20	0.2
1,3,5-Trimethylbenzene	2	20	0.2
tert-Butylbenzene	2	20	0.2
1,2,4-Trimethylbenzene	2	20	0.2
sec-Butylbenzene	2	20	0.2
1,3-Dichlorobenzene	0.5	5	.05
4-Isopropyltoluene	2	20	0.2
1,4-Dichlorobenzene	0.5	5	.05
n-Butylbenzene	2	20	0.2
1,2-Dichlorobenzene	0.5	5	.05
1,2-Dibromo-3-chloropropane (DBCP)	2	20	0.1
1,2,4-Trichlorobenzene	2	20	0.2
1,2,3-Trichlorobenzene	2	20	0.2
Naphthalene	2	20	0.1
Hexachlorobutadiene	2	20	0.2
Acetonitrile (Methyl Cyanide)	5	1000	.25
Acrolein	20	500	1
Acrylonitrile	20	50	1
2-Chloro-1,3-butadiene (Chloroprene)	10	500	0.5
3-Chloro-1-propene (Allyl Chloride)	5	50	.25
trans- + cis-1,4-Dichloro-2-butene	10	50	0.5
1,4-Dioxane	2500	12000	5
Ethyl Methacrylate	2	50	0.1
Iodomethane (Methyl Iodide)	5	50	.25
Isobutyl Alcohol (2-Methyl-1-propanol)	200	1000	1
Methacrylonitrile	5	50	.25
Methyl Methacrylate	5	50	.25
Propionitrile (Ethyl Cyanide)	5	250	.25
Vinyl Acetate	5	50	.25

TABLE 2

Standard Expiration Date Guidelines

Standard	Expiration time
Neat Chemicals	Expiration date 3 years from date opened, or supplier's assigned date.
Stock Standards (unopened ampules, commercially prepared or lab prepared)	Supplier's assigned date, or 1 year if no expiration date provided.
Opened ampules (working stocks)	
• concentration ≥ 5000 ppm	6 month expiration date.
• concentration 1000 - 5000 ppm	2 month expiration date.
• concentration < 1000 ppm	1 month expiration date.
Working Standards	
• concentration > 200 ppm (fuels)	1 month expiration date.
• concentration ≤ 200 ppm	7 day expiration date.
Internal Standard Solutions	One month expiration date.

Note: The analyst performing specific analytical procedures should use judgement and take into consideration the solution reactivity, volatility, and concentration when using standards to prepare calibration curves. Certain standards, depending on use and storage, may have shorter usable life than described in these guidelines.

TABLE 3

4-Bromofluorobenzene Characteristic Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	>95;<101% of mass 174
177	5-9% of mass 176

APPENDIX I

QC ACCEPTANCE CRITERIA

	Soil	Water
Surrogate Recovery		
Dibromofluoromethane	75-132	89-111
Toluene-d8	85-109	89-111
4-Bromofluorobenzene	49-131	79-126
Laboratory Control Sample Recovery		
1,1-Dichloroethene	73-118	62-148
Benzene	78-116	77-114
Trichloroethene	79-119	69-124
Toluene	77-118	75-118
Chlorobenzene	80-117	79-110
1,2-Dichlorobenzene	79-120	80-108
Naphthalene	57-135	64-125
Matrix Spike Recovery		
1,1-Dichloroethene	51-127	42-178
Benzene	57-121	65-138
Trichloroethene	45-127	58-146
Toluene	34-134	68-135
Chlorobenzene	37-126	71-124
1,2-Dichlorobenzene	34-131	71-121
Naphthalene	20-139	50-145

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

EPA Method 8270C

SOC-8270C
Revision 1
June 16, 1999

Approved By:

Supervisor	6/17/99 Date
QA Manager	6-17-99 Date
Laboratory Manager	6-17-99 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
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SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS
Method 8270C

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water and soil using EPA Method 8270C. This procedure may also be applicable to various miscellaneous waste samples. Table 1 indicates compounds that may be determined by this method and lists their method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) will vary depending on the instrument used and preparation method.

1.2. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. Other compounds than those listed in Table 1 may be analyzed. Refer to Section 1 of method 8270C.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. A 1.0 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration and the chromatography for this compound is poor. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, to a chemical reaction in acetone, and can undergo photochemical decomposition. N-nitroso-dimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitroso-diphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) followed by initial calibration standard(s). Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP injection time.

3.2. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.3. **Standard Curve** - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.4. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.5. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.6. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.7. **Independent Calibration Verification Standard (ICV)** - A mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction.

6.4. Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. Extracts are stored at -10°C.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2. Column: Rtx-5MS - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column. Recommended: Restek Rtx-5MS with Integra-guard, catalog #12623-124.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 2 when 1.0 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2. Stock Standard Solutions (See Table 3)

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 1000 ug/ml or more. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10°C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards or samples indicates a problem.

8.3. Internal Standard Solutions (See Table 3) - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 4 for corresponding compounds). The nominal concentration of the standard is 4000 ng/µL. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations.

8.4. GC/MS Tuning Standard (See Table 3) - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of benzidine, DDT, and pentachlorophenol, to verify injection port inertness and GC column performance. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards (See Table 3)

8.5.1. A minimum of five initial calibration standards should be prepared from stock solutions. One of the calibration standards should be at a concentration at or below the method reporting limit; the others should correspond to the range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. At least one calibration standard must be at a concentration corresponding to a sample concentration meeting project-specific data quality objectives. Each standard should contain each analyte for detection by this method. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.5.2. The daily calibration standard (CCV) is prepared at a nominal 50 ng/µL concentration from stock solutions. The CCV is prepared weekly and can be stored at 4°C ± 2°C, or as recommended by the manufacturer. The DFTPP standard may be combined with this standard (maintaining 50 ng/µL concentration) providing tuning verification and calibration verification can be done without interferences.

8.6. QC Standards (See Table 4)

8.6.1. Surrogates: Prepare a working solution in methanol containing 2-fluorophenol, phenol-d6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14 at 100 ng/µL. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2. Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest ("full list spike") at 100 ng/µL. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

Note: The spiking level of surrogate and spike may need to be adjusted according to project requirements, if dilutions are expected due to high levels of extracted components, or if a lower calibration range is used.

9. PREVENTATIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes

changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. Tune the MS as needed to result in consistent and acceptable performance (see 11.3 and 11.4).

9.4.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8270C, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water, soil, and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and screened by GC/FID (see screening SOP). Cleanup by GPC is optional.

11.1.2. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. The recommended GC/MS operating conditions:

Mass range:	35-500 amu
Scan Time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	300°C, hold until benzo[g,h,i]perylene has eluted
Injector temperature:	250-300°C
Detector interface temp:	300°C
Injector:	splitless, electron pressure control with pulse
Sample volume:	1.0 µL
Carrier gas:	helium at 35 cm/sec

NOTE: Refer to the CAS protocols for organics analyses calibration (Attachment A). The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.3. Initial Calibration

11.3.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration.

11.3.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 2 (see 8270C, Section 7.3.1 for guidance). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. To assess column performance and injection port inertness, pentachlorophenol and benzidine should be present at an acceptable level and peak tailing should not be excessive. DDT degradation should not exceed 20%. If excessive tailing, poor chromatography, or degradation of >20% is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria can not be met, the source may need cleaning, filaments replaced or other maintenance.

11.3.3. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 5 for internal standards and corresponding analytes

assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 6). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.3.4. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 6). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.3.5. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compound (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. The SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. If they are not acceptable, perform GC maintenance (see section 9.3).

11.3.6. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see below) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RSD = relative standard deviation.

$\overline{RF}$ = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

Calibration Check Compounds (CCC):

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
(N-Nitroso)-diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

11.3.7. If the % RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.3.8. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.3.9. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

11.3.9.1. The mean of the RSD values for all analytes in the calibration is $\leq$ 15%. This is easily checked using the CALEXCEL command on the Enviroquant command line. After the Excel spreadsheet is displayed, use the function wizard to average the RSDs in a designated and labeled cell.

11.3.9.2. The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.

11.3.9.3. The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

11.3.10. If all of the conditions in Section 11.3.9 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.3.8.

11.3.11. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Calibration policy.

11.4. Contunuing Calibration

11.4.1. A calibration standard, or standards, at mid-concentration (See Table 3) containing all semivolatile analytes, DFTPP, and all required surrogates, must be analyzed every 12 hours during analysis. The DFTPP must result in a mass spectrum (see 8270C, Section 7.3.1 for guidance) which meets the criteria given in Table 2. These criteria must be demonstrated during each 12 hour shift.

11.4.2. System Performance Check Compounds (SPCCs): For each daily calibration, a system performance check must be made. For each SPCC compound in the daily calibration standard, a minimum response factor of 0.050 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.3. Calibration Check Compounds (CCCs): After the system performance check, CCCs listed in Section 11.3.6 are used to check the validity of the initial calibration.

Calculate the percent drift using:

$$\%\,Drift = \frac{C_I - C_c}{C_I} \times 100$$

where:

C_I = Calibration Check Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

If the percent drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated.

This criterion must be met before sample analysis begins. If the % RSD for non-CCC compounds exceeds 30%, the analyst must determine if the response is sufficient to attain MRL for that analyte any hits for that analyte must be rerun for quantitation.

11.4.4. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quanitiation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quanitiation of subsequent analyses.

11.4.5. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.5. GC/MS Analysis

11.5.1. Evaluate FID screen and make proper dilution (See FID screening SOP).

11.5.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.5.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.5.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. Refer to Section 8.0 of Method 8270C for general QC protocol. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be

validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270C Section 8.3 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *for The Determination of Method Detecion Limits.*

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Analytical Batches and Analytical Sequences*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Table 7. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

12.3.3.1. The acceptance limits for the MS/DMS are given in Table 7. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

12.3.4. The acceptance limits for the surrogates are given in Table 7. If surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be identified.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

13.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3. The relative intensities of the characteristic ions agree within 20% of the relative intensities of these ions in the reference spectrum.

13.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the average of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks appear to represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.2. For samples containing components not associated with the calibration standards, a library search may be made of the purpose of tentative identification. Refer to method 8270C for guidance on tentatively identified compound (TIC) identification and quantification.

13.3. Quantitation and Calculations

13.3.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.4. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

13.5. Reporting

13.5.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample preap database, instrument date, and client-specified report requirements (when specified). This compilation is then transfered to a file which Excel© uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5.2. As an alternative, reports are generated using Excel© templates located in R:\SVM\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14. REFERENCES

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8270C, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

TABLE 1

SEMI-VOLATILE ORGANIC COMPOUNDS METHOD 8270C		
	–METHOD REPORTING LIMITS–	
Compound	Water (µg/L)	Soil (mg/Kg)
N-Nitrosodimethylamine	25	2
Aniline	25	1
Bis(2-chloroethyl) Ether	10	0.3
1,2-Dichlorobenzene	10	0.3
1,3-Dichlorobenzene	10	0.3
1,4-Dichlorobenzene	10	0.3
Bis(2-chloroisopropyl) Ether	10	0.3
N-Nitrosodi-n-propylamine	10	0.3
Hexachloroethane	10	0.3
Nitrobenzene	10	0.3
Isophorone	10	0.3
Bis(2-chloroethoxy)methane	10	0.3
1,2,4-Trichlorobenzene	10	0.3
Naphthalene	10	0.3
4-Chloroaniline	10	0.3
Hexachlorobutadiene	10	0.3
2-Methylnaphthalene	10	0.3
Hexachlorocyclopentadiene	10	0.3
2-Chloronaphthalene	10	0.3
2-Nitroaniline	25	2
Dimethyl Phthalate	10	0.3
Acenaphthylene	10	0.3
3-Nitroaniline	25	2
Acenaphthene	10	0.3
Dibenzofuran	10	0.3
2,4-Dinitrotoluene	10	0.3
2,6-Dinitrotoluene	10	0.3
Diethyl Phthalate	10	0.3
4-Chlorophenyl Phenyl Ether	10	0.3
Fluorene	10	0.3

TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS METHOD 8270C		
	–METHOD REPORTING LIMITS–	
Compound	Water (µg/L)	Soil (mg/Kg)
4-Nitroaniline	25	2
N-Nitrosodiphenylamine	10	0.3
4-Bromophenyl Phenyl Ether	10	0.3
Hexachlorobenzene	10	0.3
Phenanthrene	10	0.3
Anthracene	10	0.3
Di-n-butyl Phthalate	10	0.3
Fluoranthene	10	0.3
Pyrene	10	0.3
Butylbenzyl Phthalate	10	0.3
3,3'-Dichlorobenzidine	25	2
Benz(a)anthracene	10	0.3
Bis(2-ethylhexyl) Phthalate	10	0.3
Chrysene	10	0.3
Di-n-octyl Phthalate	10	0.3
Benzo(b)fluoranthene	10	0.3
Benzo(k)fluoranthene	10	0.3
Benzo(a)pyrene	10	0.3
Indeno(1,2,3-c,d)pyrene	10	0.3
Dibenz(a,h)anthracene	10	0.3
Benzo(g,h,i)perylene	10	0.3
Phenol	10	0.3
2-Chlorophenol	10	0.3
Benzyl Alcohol	10	0.3
2-Methylphenol	10	0.3
3- and 4-Methylphenol♦	10	0.3
2-Nitrophenol	10	0.3
2,4-Dimethylphenol	10	0.3
Benzoic Acid	25	2
2,4-Dichlorophenol	10	0.3

♦ Co-eluting compounds

TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS METHOD 8270C		
	–METHOD REPORTING LIMITS–	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
4-Chloro-3-methylphenol	10	0.3
2,4,6-Trichlorophenol	10	0.3
2,4,5-Trichlorophenol	10	0.3
2,4-Dinitrophenol	25	2
4-Nitrophenol	25	2
2-Methyl-4,6-dinitrophenol	25	2
Pentachlorophenol	25	2
2-Picoline	10	-
N-Nitrosodiethylamine	10	0.3
Methyl Methanesulfonate	10	0.3
N-Nitrosoethylmethylamine	10	0.3
Pentachloroethane	10	-
Acetophenone	10	0.3
N-Nitrosopyrrolidine	10	0.3
N-Nitrosomorpholine	10	0.3
N-Nitrosopiperidine	10	0.3
O,O,O-Triethyl Phosphorothioate	10	0.3
2,6-Dichlorophenol	10	0.3
Hexachloropropene	10	0.3
N-Nitrosodi-*n*-butylamine	10	0.3
p-Phenylenediamine	10	0.3
Safrole	10	0.3
1,2,4,5-Tetrachlorobenzene	10	0.3
Isosafrole	10	2
1,3-Dinitrobenzene	10	0.3
Pentachlorobenzene	10	0.3
1-Naphthylamine	10	0.3
2-Naphthylamine	10	0.3
2,3,4,6-Tetrachlorophenol	10	0.3
Diphenylamine	10	0.3

TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS METHOD 8270C		
	–METHOD REPORTING LIMITS–	
Compound	Water (µg/L)	Soil (mg/Kg)
1,3,5-Trinitrobenzene	25	2
Phenacetin	50	2
4-Aminobiphenyl	10	0.3
4-Nitroquinoline N-Oxide	10	-
Total Aramite	50	-
3,3'-Dimethylbenzidine	25	2
7,12-Dimethylbenz(a)anthracene	10	0.3
Hexachlorophene	5,000	-
3-Methylcholanthrene	10	0.3
N,N-Dimethyl-1-phenethylamine	10	-
2-Acetylaminofluorene	10	0.3
o-Toluidine	10	0.3
Ethyl Methanesulfonate	10	0.3
1,4-Naphthoquinone	10	0.3
5-Nitro-*o*-toluidine	10	0.3
p-Dimethylaminoazobenzene	10	0.3
Pentachloronitrobenzene	50	2
Methapyrilene	100	4
Chlorobenzilate	10	0.3
2-sec-Butyl-4,6-Dinitrophenol	25	1
Diallate	10	0.3
Dimethoate	10	0.3
Disulfoton	10	0.3
Famphur	10	0.3
Isodrin	10	0.3
Kepone	100	4
Methyl Parathion	10	0.3
Parathion	10	0.3
Phorate	10	0.3
Pronamide	10	0.3
Thionazine	25	2

TABLE 2
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

Alternate tuning criteria (from Method 525.2 or CLP OLM03.1) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

TABLE 3
8270 STANDARDS

CALIBRATION

Recommended: AccuStandard catalog #(or equivalent from other vendors*):

CLP-HC-BN-R	1 ml x 2 2000 ppm BN mix
CLP-HC-A-PAK	1 ml x 2 2000 ppm Acid composite mix
Z-014E-R3	1 ml x 2 2000 ppm Composite 3 mix
M-8270-SS-PAK	1 ml x 5 4000 ppm Surrogates mix
Z-014J	1 ml x 5 4000 ppm Internal standards mix
M-625C	1 ml x 3 25000 ppm DFTPP
Z-014F	1 ml x 2 2000 ppm Benzidines mix

Prepare 5 ml of each calibration point from a new unopened ampule.
Calibration curve: 5 ppm, 10 ppm, 20 ppm, 50 ppm, 80 ppm, 120 ppm, 160 ppm, and 200 ppm.
Add internal standard when curve is prepared.
Place ~ 100 µl in autosample vial insert, run, and discard after verifying data is acceptable.
Store in 5 ml amber mininert vials (2-5 ml vials for 10 ml) at -10°C. Expiration is 1 year from date prepared.
Order more solutions when down to one unopened ampule.

ICV

Two ICV standards are prepared from the following Ultra stock standards (all 1 ml x 2 2000ppm, expect US-112 which is 1 ml x 2 500ppm), or equivalent from other vendors*:

ICV-1: US-104, US-106, US-107, US-110, US-111, US-113, US-114, US-115, US-116, US-117, US-118, US-119, US-120, Carbazole(Supelco), and CLP-031R2 (Accustandard).

ICV-2: US-112, US-113-1, US-103N

Add 10 µl internal standard when ampule is opened.
Place ~ 100 µl in autosample vial insert, run, and discard after verifying data is acceptable.
Store open ampule in 1 ml mininert vial. Expiration is 1 week after ICV was prepared.
Order when down to one unopened ampule.

CCV & TUNE

Use the same solutions that were used for the calibration curve and the following:

AccuStandard (recommended) catalog #(or equivalent from other vendors*):

P-0295-20X	1 ml x 1 2000 ppm DDT

Prepare 1 ml of 50 ppm 8270 CCV standard, place in autosample vial and cap with red crimp top.
Expiration date is 1 week after CCV was prepared.

RECAP AND STORE IMMEDIATELY AFTER INJECTING

Store remaining stock solutions in 1 ml amber mininert vial. Expiration date is six months after ampule is opened. Order when down to one unopened ampule.

* Vendor must be A2LA and/or ISO9000 certified.

TABLE 4
QC Standards

8270 SURROGATE
100/150ppm

****Each time you make it, Order:**

Supelco
4 - 20006173 BN Surrogate Standards (*Custom mix*)
3 - 20006175 Acid Surrogate Standards (*Custom mix*)

RECIPE***

Parent	Initial Concentration	Aliquot	Final volume	Final Concentration	Solvent
20006173	5000ug/ml	20.0mls	****1000.0mls	100ug/ml	MeOH
20006175	10000ug/ml	15.0mls		150ug/ml	MeOH

Standards expiration: Unopened=6mo. from prep date. Opened=3mo. after opening or original 6mo., whichever comes FIRST. When breaking the seal on an unopened bottle, write the new expiration date on the bottle and in the standards logbook with your initials and date opened.
Absolutely No expired standards stored or left in standards freezer.
* *Custom mixes must be accompanied by a quote sheet.*
** *Ordered number reflects minimum amount of backups needed at all times.*
*** *To reduce wasted standards, vary amounts made to reflect the workload for this test.*
**** *Split final volume into 4-250ml bottles.*

8270 MATRIX SPIKE
100ppm

***Each time you make it, Order:**

Supelco
2 - 20006268 CLP Semivol. Calib. mix (*Custom mix*)
**20005887 *Custom Mix*

RECIPE

Parent	Initial Concentration	Aliquot	Final volume	Final Concentration	Solvent
20006268	1000ug/ml	10.0mls	***100.0mls	100ug/ml	MeOH
20005887	1000ug/ml	10.0mls		100ug/ml	MeOH

Standards expiration: Unopened=6mo. from prep date. Opened=3mo. after opening or original 6mo., whichever comes FIRST. When breaking the seal on an unopened bottle, write the new expiration date on the bottle and in the standards logbook with your initials and date opened.
Absolutely No expired standards stored or left in standards freezer.
* *Custom mixes must be accompanied by a quote sheet.*
** *When there are only two left, order a minimum quantity of 8. This saves $155.00 per unit.*
*** *Split final volume into 2-50ml bottles.*

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr.)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr.)
4-Methylphenol	Nitrobenzene	Hexachlorocyclo-pentadiene
N-Nitrosodimethylamine	Nitrobenzene-d_5 (surr.)	1-Naphthylamine
N-Nitroso-di-n-propyl-amine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodibutylamine	2-Nitroaniline
Phenol-d_6 (surr.)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tetra-chlorophenol
		2,4,6-Tribromo-phenol (surr.)
		2,4,6-Trichloro-phenol
		2,4,5-Trichloro-phenol

(surr.) = surrogate

TABLE 5 cont.

Phenanthrene-d(10)	Chrysene-d(12)	Perylene-d(12)
4-Aminobiphenyl	Benzidine	Benzo(b)fluor-anthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)-perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methyl-phenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h) anthracene
Fluoranthene	p-Dimethylaminoazobenzene	7,12-Dimethylbenz-(a)anthracene
Hexachlorobenzene	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Terphenyl-d,(14)(surr.)	Indeno(1,2,3-cd) pyrene
Pentachlorophenol		3-Methylchol-anthrene
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr.) = surrogate

TABLE 6

Characteristic Ions For Semivolatile Compounds

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75(a)	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d(4)I.S.)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,88,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	62,44,45,74
Thiophenol (Benzenethiol)	7.42	110	110,66,109,84
Methyl methanesulfonate	7.48	80	80,79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	54,98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodie thylamine	8.70	102	102,42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,108,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	110,79,95,109,140
Ethyl methanesulfonate	9.62	79	79,109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d(8)I.S.)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	99,155,127,81,109
Diethyl sulfate	11.37	139	139,45,59,99,111, 125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	107,108,77,79,90
Hexachloropropene	12.45	213	213,211,215,

TABLE 6 cont.

Hexachlorocyclopentadiene	12.60	237	117,106,141 235,272
N-Nitrosopyrrolidine	12.65	100	100,41,42,68,69
Acetophenone	12.67	105	71,105,51,120
4-Methylphenol	12.82	107	107,108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	106,107,77,51,79
3-Methylphenol	12.93	107	107,108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,114,55,56,41
1,4-Phenylenediamine	13.62	108	108,80,53,54,52
1-Chloronaphthalene	13.65(a)	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	106,141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	65	63,89
Phthalic anhydride	14.62	104	104,76,50,148
o-Anisidine	15.00	108	80,108,123,52
3-Nitroaniline	15.02	38	108,92
Acenaphthene-d(10)I.S.)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	162,164,126,98,63
4-Chloroaniline	15.50	127	127,129,65,92
Isosafrole	15.60	162	162,131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	121,122,94,77,
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00(a)	143	115,116
1,4-Naphthoquinone	16.23	158	58,104,102,76, 50,130
p-Cresidine	16.45	122	122,94,137,77,93
Dichlorovos	16.48	109	109,185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	120,135,134,91,77
N-Nitrosodibutylamine	16.73	84	84,57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	110,81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	110,81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	162,162,104,77, 103,135
Hexamethyl phosphoramide	17.33	135	135,44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	92,127,129,65,39
Diphenylamine	17.54(a)	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	216,214,179,108, 143,218
1-Naphthylamine	18.20	143	143,115,89,63
1-Acetyl-2-thiourea	18.22	118	43,118,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	174,145,173,146, 132,91
2,4,5-Trichlorophenol	18.47	196	196,198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	84,133,161,162
Pentachlorophenol	19.25	266	264,268

TABLE 6 cont.

5-Nitro-o-toluidine	19.27	152	77,152,79,106,94
Thionazine	19.35	107	96,107,97,143, 79,68
4-Nitroaniline	19.37	138	138,65,108,92, 80,39
Phenanthrene-d(10) (i.s.)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	168,75,50,76, 92,122
Mevinphos	19.90	127	127,192,109,67, 164
Naled	20.03	109	109,145,147,301, 79,189
1,3-Dinitrobenzene	20.18	168	168,76,50,75, 92,122
Diallate (cis or trans)	20.57	86	86,234,43,70
1,2-Dinitrobenzene	20.58	168	168,50,63,74
Diallate (trans or cis)	20.78	86	86,234,43,70
Pentachlorobenzene	21.35	250	250,252,108,248, 215,254
5-Nitro-o-anisidine	21.50	168	168,79,52,138, 153,77
Pentachloronitrobenzene	21.72	237	237,142,214,249, 295,265
4-Nitroquinoline-1-oxide	21.73	174	174,101,128,75, 116
Di-n-butyl phthalate	21.78	149	150,104,2,3,4
6-Tetrachlorophenol	21.88	232	232,131,230,166, 234,168
Dihydrosaffrole	22.42	135	135,64,77
Demeton-o	22.72	88	88,89,60,61,115, 171
Fluoranthene	23.33	202	101,203 1,3,
5-Trinitrobenzene	23.68	75	75,74,213,120,91, 63
Dicrotophos	23.82	127	127,67,72,109, 193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	306,43,264,41, 290
Bromoxynil	23.90	277	277,279,88,275, 168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	127,192,67,97,109
Phorate	24.10	75	75,121,97,93,260
Sulfallate	24.23	188	188,88,72,60,44
Demeton-s	24.30	88	88,60,81,89,114, 115
Phenacetin	24.33	108	180,179,109,137, 80
Dimethoate	24.70	87	87,93,125,143, 229
Phenobarbital	24.70	204	204,117,232,146, 161
Carbofuran	24.90	164	164,149,131,122
Octamethyl pyrophosphoramide	24.95	135	135,44,199,286, 153,243
4-Aminobiphenyl	25.08	169	169,168,170,115

TABLE 6 cont.

Dioxathion	25.25	97	97,125,270,153
Terbufos	25.35	231	231,57,97,153,103
a Dimethylphenylamine	25.43	58	58,91,65,134,42
Pronamide	25.48	173	173,175,145,109, 147
Aminoazobenzene	25.72	197	92,197,120,65,77
Dichlone	25.77	191	191,163,226,228, 135,193
Dinoseb	25.83	211	211,163,147,117, 240
Disulfoton	25.83	88	88,97,89,142,186
Mexacarbate	26.02	165	165,150,134,164, 222
4,4'-Oxydianiline	26.08	200	200,108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	199,152,141,169, 151
Phosphamidon	26.85	127	127,264,72,109, 138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	231,185,41,193, 266
Methyl parathion	27.03	109	109,125,263,79,93
Carbaryl	27.17	144	144,115,116,201
Dimethylaminoazobenzene	27.50	225	225,120,77,105, 148,42
Propylthiouracil	27.68	170	170,142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d(12) I.S.)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	173,125,127,93, 158
Kepone	28.18	272	272,274,237,178, 143,270
Fenthion	28.37	278	278,125,109,169, 153
Parathion	28.40	109	109,97,291,139, 155
Anilazine	28.47	239	239,241,143,178, 89
Bis(2-ethylhexyl)phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	212,106,196,180
Carbophenothion	28.58	157	157,97,121,342, 159,199
5-Nitroacenaphthene	28.73	199	199,152,169,141, 115
Methapyrilene	28.77	97	97,50,191,71
Isodrin	28.95	193	193,66,195,263, 265,147
Captan	29.47	79	79,149,77,119,117
Chlorfenvinphos	29.53	267	267,269,323,325, 295
Crotoxyphos	29.73	127	127,105,193,166
Phosmet	30.03	160	160,77,93,317,76
EPN	30.11	157	157,169,185,141, 323
Tetrachlorvinphos	30.27	329	109,329,331,79, 333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	223,167,195

TABLE 6 cont.

Barban	30.83	222	222,51,87,224, 257,153
Aramite	30.92	185	185,191,319,334, 197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	283,285,202,139, 253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	251,139,253,111, 141
Fensulfothion	31.87	293	293,97,308,125, 292
Ethion	32.08	231	231,97,153,125, 121
Diethylstilbestrol	32.15	268	268,145,107,239, 121,159
Famphur	32.67	218	218,125,93,109, 217
Tri-p-tolyl phosphateb	32.75	368	368,367,107,165, 198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d(12)I.S.)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	256,241,239,120
5,5-Diphenylhydantoiin	33.40	180	180,104,252,223, 209
Captafol	33.47	79	79,77,80,107
Dinocap	33.47	69	69,41,39
Methoxychlor	33.55	227	227,228,152,114, 274,212
2-Acetylaminofluorene	33.58	181	181,180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	231,266,268,140, 195
3,3'-Dimethoxybenzidine	34.47	244	244,201,229
3-Methylcholanthrene	35.07	268	268,252,253,126, 134,113
Phosalone	35.23	182	182,184,367,121, 379
Azinphos-methyl	35.25	160	160,132,93,104, 105
Leptophos	35.28	171	171,377,375,77, 155,379
Mirex	35.43	272	272,237,274,270, 239,235
Tris(2,3-dibromopropyl)phosphate	35.68	201	137,201,119,217, 219,199
Dibenz(a,j)acridine	36.40	279	279,280,277,250
Mestranol	36.48	277	277,310,174,147, 242
Coumaphos	37.08	362	362,226,210,364, 97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279 1,2 4,
5 Nibenzopyrene	41 60	302	302,151,150,300
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	162,135,105,77
Hexachlorophene	47.98	196	196,198,209,211, 406,408
Aldrin	--	66	263,220
Aroclor-1016	--	222	260,292
Aroclor-1221	--	190	224,260

TABLE 6 cont.

Aroclor-1232	--	190	224,260
Aroclor-1242	--	222	256,
Aroclor-1248	--	292	362,32
Aroclor-1254	--	292	362,326
alpha-BHC	--	183	181,109
beta-BHC	--	181	183,109
delta-BHC	--	183	181,109
gamma-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr.)	--	172	171
2-Fluorophenol (surr.)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d(5)surr.)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d(6) (surr.)	--	99	42,71
Terphenyl-d(14) (surr.)	--	244	122,212
2,4,6-Tribromophenol (surr.)	--	330	332,141
Toxaphene	--	159	231,233

I.S. = internal standard.

surr. = surrogate.

a) Estimated retention times.

b) Substitute for the non-specific mixture, tricresyl phosphate.

Table 7
QC Acceptance Criteria For Surrogate, MS, LCS

SURROGATE SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
Nitrobenzene-d_5	32-123	21-115
2-Fluorobiphenyl	42-122	34-117
p-Terphenyl-d_{14}	21-167	43-159
Phenol-d_6	22-118	30-104
2-Fluorophenol	7-105	27-106
2,4,6-Tribromophenol	31-141	18-140

LABORATORY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Compound	Water	Soil/Sediment
Phenol	37-102	32-97
2-Chlorophenol	38-108	32-105
1,4-Dichlorobenzene	51-98	29-100
N-Nitrosodi-n-propylamine	43-114	26-112
1,2,4-Trichlorobenzene	42-113	31-109
4-Chloro-3-methylphenol	39-120	31-121
Acenaphthene	50-114	46-105
4-Nitrophenol	15-147	21-133
2,4-Dinitrotoluene	55-123	54-114
Pentachlorophenol	34-126	38-107
Pyrene	49-125	43-129

MATRIX SPIKE PERCENT RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Matrix Spike Compound	Water	Soil/Sediment
Phenol	31-96	29-92
2-Chlorophenol	37-104	35-90
1,4-Dichlorobenzene	39-100	30-82
N-Nitrosodi-n-propylamine	37-107	19-108
1,2,4-Trichlorobenzene	35-113	33-90
4-Chloro-3-methylphenol	39-118	35-108
Acenaphthene	52-102	33-107
4-Nitrophenol	15-157	24-119
2,4-Dinitrotoluene	51-114	40-109
Pentachlorophenol	18-129	18-108
Pyrene	28-129	24-130

Attachment A

CAS Organics Analysis Calibration Policy and Procedure

Columbia Analytical Services, Inc.
An Employee-Owned Company

MEMORANDUM

DATE: May 19, 1999

TO: Department Managers, Kelso Organics

FROM: Joe Wiegel

SUBJ: **INITIAL AND CONTINUING CALIBRATIONS FOR ORGANICS, DRAFT REVISION NO. 9**

This memo states our policy on performing and evaluating initial and continuing calibrations for organic analyses. The guidelines stated in the memo are designed as default specification. Unless stated in standard operating procedures and prescribed in the determinative method, this memo states our standard operating procedure for calibrations. This policy is consistent with EPA Method 8000B, SW-846, Third Edition, Update III.

1. General Calibration Guidelines

   1.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

   1.2. Calibrations for organic analyses must contain a minimum of five concentrations.

   1.3. The method reporting limit (MRL) must be supported by the calibration, typically as the low point in the calibration.

   1.4. The complete calibration (i.e., all initial calibration (ICAL) levels and the independent calibration verification (ICV) standard) must be analyzed prior to analysis of field or QC samples.

   1.5. A calibration may not be interrupted by a maintenance event.

   1.6. A calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated.

   1.7. Calibration points may be dropped at the endpoints of the curve as long as conditions 1.1, 1.2 and 1.3 are met.

1.8. Calibration points may not be dropped from the interior of a curve unless a catastrophic error (e.g., gross dilution error, missing internal standards, injection malfunction, etc.) is accounted for in a nonconformity and corrective action report (NCAR). In these circumstances, all the analytes in that calibration standard must be dropped from the calibration curve as corrective action.

1.9. Analysis of all field and QC samples must be preceded by an acceptable ICAL and ICV, or must be preceded by an acceptable CCV that verifies the ICAL.

2. Evaluation Guidelines for Initial Calibrations

2.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

2.2. Average response factor (RF*ave*) is the preferred calibration technique because linearity through the origin is assumed. As such, this technique allows the analyst to perform a more intuitive assessment of data below the lowest calibration standard. However, RF*ave* may not always be the best fit of calibration data. The analyst should use prior knowledge of the instrument, analyte response, and an assessment of the calibration data in determining whether RF*ave* is appropriate.

2.2.1. Acceptance criteria for RF*ave*:

GC and HPLC

2.2.1.1. Relative standard deviation (RSD) equal to or less than 20% for all compounds.

GC/MS

2.2.1.2. System performance and calibration check compounds (SPCC and CCC) must meet method criteria.

2.2.1.3. Relative standard deviation (RSD) equal to or less than 15% for all compounds.

Allowable Exceptions

2.2.1.4. Some analytes are recognized as marginal performing compounds. Typically, these are analytes that are not expected to meet the primary evaluation criteria due to the chemical nature of the compound. Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. The list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be indicated in the analytical SOP. Acceptance criteria for RF*ave* for these compounds are as follow:

GC and HPLC Procedures

2.2.1.4.1. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%

2.2.1.4.2. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 20%.

2.2.1.4.3. The RSD for marginal performing compounds must be less than or equal to 40%.

GC/MS Procedures

2.2.1.4.4. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

2.2.1.4.5. The average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.1.4.6. The RSD for individual analytes not designated as marginal performing compounds in the analytical SOP must be less than or equal to 15%.

2.2.1.4.7. The RSD for marginal performing compounds must be less than or equal to 30%.

2.2.2. A calibration that has been processed using RF*ave* that does not meet the criteria described above may be used to report non-detected analytes. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. The following criteria apply:

2.2.2.1. For GC and HPLC procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 20%.

2.2.2.2. For GC/MS procedures, the average RSD for the calibration, using all analytes in the method, must be less than or equal to 15%.

2.2.2.3. Adequate sensitivity to detect and confirm the analyte at the MRL must be demonstrated by the lowest calibration standard.

2.2.2.4. All confirmed detections for analytes exceeding RSD criteria in the ICAL, regardless of concentration, must be reanalyzed to ensure accurate quantification following recalibration of the instrument.

2.3. When curves are used, least squares and quadratic fits are permissible. These fits will not be forced through the origin. As such, it may become impractical to report estimated concentrations in samples below the lowest standard of calibration. When used, the analyst must be trained on the significance of the Y-intercept when using curve fits. Specifically, the analyst must understand that very low responses may

quantify to false positives depending on where the curve intersects the Y axis. Acceptance criteria for these fits are as follow:

2.3.1. Least Squares: $R \geq 0.995$ or $R^2 \geq 0.990$

2.3.2. Quadratic: $COD \geq 0.990$

2.3.3. If a least squares fit is used, the curve must contain a minimum of five (5) calibration levels.

2.3.4. If a quadratic fit is used, the curve must contain a minimum of six (6) calibration levels and must be continuous along the function (i.e., it must consist of consecutive increasing or consecutive decreasing numerical values).

2.4. Because calibration curves can not be forced through the origin, the analyst should evaluate the effect the y-intercept will have on quantifying detections at or below the lowest standard of calibration. This evaluation should involve extrapolating the curve to a level of one half the lowest standard (using one half the area or area ratio). If the result is positive and less than the MRL, the curve may be used. If the result is equal to or less than zero, or if the result is equal to or greater than the MRL, the curve fit is not to be used.

2.5. The calibration will be verified by an ICV standard (i.e., a second source standard) prior to analysis of field or QC samples. The ICV should be analyzed each time the calibration curve is analyzed and on each instrument that is calibrated. Acceptance criteria for all techniques (GC, HPLC and GC/MS) are as follow:

2.5.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

2.5.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

2.5.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±30% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

2.5.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. ICV acceptance criteria on these analytes is ±30% for GC and HPLC procedures and ±40% for GC/MS procedures.

3. Evaluation Criteria: Continuing Calibration Verification (CCV) Standards

3.1. Criteria specified in the determinative method or by project specific quality assurance plans take precedence over these guidelines.

3.2. For calibrations using RF*ave*, CCV Standards are evaluated based on % Difference (%Dif) of the response factor. %Dif is calculated as:

$$\%\,Dif = \frac{RFv - RFave}{RFave} \times 100$$

Where:

RFv is the response factor (also relative response factor) or calibration factor from the verification standard and ***RFave*** is the average response factor or calibration factor from the calibration.

3.3. For calibrations using Least Squares or Quadratic fits, CCV Standards are evaluated based on % Drift (%Drft) of the measured value compared to the expected value. %Drft is calculated as:

$$\%\,Drft = \frac{Measured\ concentration - Expected\ concentration}{Expected\ concentration} \times 100$$

3.4. CCV Acceptance Criteria:

GC and HPLC Procedures

3.4.1. The average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.2. For individual analytes, the maximum allowable %Dif or %Drft is ±15% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.3. For multi-component pesticides and PCB Aroclors, the maximum allowable %Drft is ±15% for the average result of the quantitation. There is no criteria placed on individual peaks used to quantitate the multi-component analyte.

3.4.4. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±30%.

GC/MS Procedures

3.4.5. Method specified CCC and SPCC criteria must be met when these compounds are included in the analysis.

3.4.6. The average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft is based on the absolute value of all analytes in the calibration irrespective of the analyte list being reported.

3.4.7. For individual analytes, the maximum allowable %Dif or %Drft is ±20% except as noted below under allowable exceptions. Samples with confirmed detections of analytes that do not meet this criteria must be reanalyzed.

3.4.8. Allowable Exceptions: Standard operating procedures (SOP) may designate a list of marginal performing compounds that are included in the analysis. This list is limited to 15% of the complete analyte list for the procedure (calculated as the total number of analytes times 0.15, rounded down to the nearest integer). Analytes designated as marginal performing compounds must be so indicated in the analytical SOP. CCV acceptance criteria on these analytes is ±40%.

3.5. Non-detected analytes can be reported from analytical sequences that contained CCVs that do not pass acceptance criteria. However, this is generally considered a temporary measure and is allowed only for reporting the absence of a target analyte. All positive and confirmed detections, regardless of analyte concentration, method detection limit or method reporting limit, must be reanalyzed following recalibration of the instrument to assure accurate quantification. This includes reanalysis of QA/QC samples containing spiked amounts of target analyte. The following criteria apply:

3.5.1. For GC and HPLC procedures, the average %Dif or %Drft must be ±15% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.2. For GC/MS procedures, the average %Dif or %Drft must be ±20% for the calibration. The calculation of average %Dif or %Drft must be based on the absolute value all analytes in the calibration irrespective of the analyte list being reported.

3.5.3. The analysis must demonstrate adequate sensitivity to detect and confirm the analyte at the MRL. Therefore, all analytes being reported to the client can not exhibit a %Dif or %Drft greater than ±40%. For external calibrations, this criteria applies to both preceding and concluding CCVs.

3.5.4. All confirmed detections for analytes that fail acceptance criteria in the CCV, regardless of concentration, must be reanalyzed to ensure accurate quantification. This criteria applies to all samples and QA/QC samples analyzed in the sequence.

3.6. CCV standards must be analyzed at the start of each analytical sequence (except when the sequence is initiated with an ICAL and ICV).

3.7. Two sequential analyses of a CCV at the beginning of an analytical sequence can be performed in an effort to prime the instrument for analysis. Routine analysis of two or

more sequential CCV standards throughout the analytical sequence in an effort to ensure continuation of the sequence is not permitted.

3.8. CCV standards are analyzed at the following frequency:

External Standard Calibrations

3.8.1. CCV standards should be analyzed after every 10 injections of field and QC samples or every 12 hours, which ever is more frequent.

3.8.2. Samples with confirmed detections must be bracketted by acceptable CCV standards.

3.8.3. When a closing CCV standard is not acceptable, corrective action must be taken. A new CCV standard may be prepared and analyzed to demonstrate degradation of the standard as the cause of a CCV outlier. In this case, instument stability will be verified and samples analyzed prior to this CCV can be reported. CCV standards that are reinjected after minor instrument maintenance (e.g., injection port maintenance, column bake-out, installation of a new trap, etc.) do not verify instrument stability. Samples analyzed prior to this CCV must be reanalyzed.

Internal Standard Calibrations

3.8.4. The analysis sequence must be initiatied with an acceptable instrument tune (for GC/MS) and an acceptable CCV. The last injection or analysis in the sequence must be started within 12 hours from the time of sequence initiation.

3.8.5. Internal standard response in the CCV should be within 0.5 - 2 times the response observed in the mid point calibration standard in the curve. Failure to meet this criteria should prompt the analyst to check the internal standard solution for possible degradation or concentration, or may indicate the need for instrument maintenance.

3.8.6. Internal standard response in each sample must be within 0.5 - 2 times the response observed in the initial CCV standard of the analytical sequence (or the mid point calibration standard in the curve if the analytical sequence began with an ICAL). Perform reanalysis or dilutions to assess the effect of matrix interferences on internal standard responses.

STANDARD OPERATING PROCEDURE

for

ULTRASONIC EXTRACTION

SOP No.: EXT-3550

Revision: 5

July 7, 1999

Approved by: _____ 7/7/99
Supervisor — Date

_____ 7-7-99
QA Manager — Date

_____ 7-7-99
Laboratory Manager — Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

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ULTRASONIC EXTRACTION

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in EPA Method 3550B for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sediments, sludges, and wastes.

1.2. This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures. The low concentration method (individual organic components of < 20 mg/Kg) uses a larger sample size and a more rigorous extraction procedure.

2. SUMMARY OF METHOD

2.1. A sample is mixed with anhydrous sodium sulfate to form a free flowing powder. The sample is solvent extracted three times using ultrasonic extraction. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent. The solvents used for extraction and concentration are dependent on the analysis being performed. A portion of the extract is removed for cleanup and/or analysis.

2.2. It is highly recommended that the extracts be cleaned up prior to analysis. Refer to appropriate cleanup SOPs and methods.

3. INTERFERENCES

3.1. Phthalate esters can pose difficulties when performing sample extractions for organochlorine pesticides, PCBs, and other semi-volatile organics. Phthalates are easily extracted or leached from materials containing plastics during laboratory operations. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials.

3.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. All apparatus must be cleaned prior to use on individual samples.

3.3. Refer to SW-846 Method 3500 for additional discussion of interferences. Additional cleanup procedures are described in the applicable CAS SOP.

4. SAFETY

4.1. All appropriate safety precautions for handling solvents, reagents, and samples must be taken when performing this procedure. This includes the use of protective equipment (safety glasses, lab coats, gloves, etc.) and use of correct glassware handling practices.

4.2. Chemicals, reagents, standards, and samples must be handled as described in CAS safety policies, approved methods, and in MSDSs where available. Refer to the specific analytical method and the CAS Safety Manual for guidance.

5. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

6. APPARATUS AND MATERIALS

6.1. Apparatus for grinding dry waste samples.

6.2. Ultrasonic preparation - A horn type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used. The horn should be tuned prior to sample extraction. (See Attached Tuning Procedure - Appendix A)

Ultrasonic Disrupter - The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturers instructions for preparing the disrupter for extraction of samples with low and medium/high concentration. Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

6.3. Sonabox - Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.4. Pasteur glass pipets - 1 ml, disposable.

6.5. Beakers - 250 or 400 ml.

6.6. Vacuum filtration apparatus.

6.7. Drying funnel - modified funnel with Pyrex glass wool at bottom.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 ml of acetone followed by 50 ml of elution solvent prior to packing the column with adsorbent.

6.8. Kuderna-Danish (K-D) apparatus.

6.8.1. Concentrator tube - 10 ml, graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

6.8.2. Evaporation flask - 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

6.8.3. Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

6.8.4. Springs or clips for attaching concentrator tubes.

6.9. Boiling chips - Pre-cleaned via Soxhlet extraction, approximately 10/40 mesh (silicon carbide or equivalent).

6.10. Water bath - Heated, with concentric ring cover, capable of temperature control (± 5-C). The batch should be used in a hood.

6.11. Balance - Top loading, capable of accurately weighing to the nearest 0.01 g.

6.12. Vials - 2 ml, for GC autosampler, with Teflon lined screw caps or crimp tops.

6.13. Glass scintillation vials - 20 ml, with Teflon lined screw caps.

6.14. Spatula - Stainless steel or Teflon.

6.15. Syringes or Eppendorf-type pipets - appropriate size for QC spiking.

7. REAGENTS

7.1. Pesticide grade inorganic chemicals shall be used in all tests. Other grades may be used, provided it is first confirmed that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination or introducing interferences.

7.2. Organic-free reagent water. This may be deionized water or tap water if it has been determined to be free of interferences and trace levels or target analytes.

7.3. Sodium sulfate (powdered, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray or crucible, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

7.4. Extraction solvents.

7.4.1. Low concentration soil/sediment and aqueous sludge samples shall be extracted using a solvent system that gives optimum, reproducible recovery for the matrix/analyte combination to be measured.

7.4.2. Methylene chloride:Acetone, CH_2Cl_2:CH_3COCH_3 (1:1, v:v). Pesticide quality or equivalent. Other solvent ratios can be used if acceptable method performance is demonstrated.

7.4.3. Methylene chloride, CH_2Cl_2. Pesticide quality or equivalent.

7.4.4. Hexane, C_6H_{14}. Pesticide quality or equivalent.

7.5. Exchange solvents.

7.5.1. Hexane, C_6H_{14}. Pesticide quality or equivalent.

7.5.2. Propanol, $(CH_3)_2CHOH$. Pesticide quality or equivalent.

7.5.3. Cyclohexane, C_6H_{12}. Pesticide quality or equivalent.

7.5.4. Acetonitrile, CH_3CN. Pesticide quality or equivalent.

7.5.5. Methanol, CH_3OH. Pesticide quality or equivalent.

8. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the determinative SOP and method. Also, refer to the introductory material in SW-846, Organic Analysis, Sec. 4.

9. PREVENTIVE MAINTENANCE

9.1. Routine cleaning of the extraction apparatus is necessary, including all parts exposed to contact with samples, especially ultrasonic horns cells.

9.2. The ultrasonic horn must be tuned prior to use. Horn tip and tuning procedures are posted in the extraction area. Proper operation of the horn is critical in achieving good method performance. Refer to the manufacturer's specifications in Appendix A.

10. PROCEDURE

10.1. Record all extraction and sample information on the applicable benchsheet. To assist the analyst, a brief description of the procedure is given on the back side of the benchsheet. See Attachment A.

10.2. Subsampling

10.2.1. Sediment/soil samples - Decant and discard any water layer on a the sample.

Note: For certain sediment samples, the water layer may be of interest. If so, or if the water layer is significant, the supervisor and project chemist should determine any special handling procedures. If sample contains a significant portion of water, water is pulled off and extracted.

10.2.2. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

10.2.3. Waste samples - Samples consisting of multi phases have each phase extracted separately. Oil phases are pulled off, usually diluted in hexane or iso-octane and extracted. This procedure is for solids only.

10.2.4. Dry waste samples amenable to grinding - Grind or otherwise subdivide the waste so that it passes through a 1 mm sieve.

10.2.5. Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling these difficult matrices.

10.3. Determination of sample % dry weight - In certain cases, sample results are desired based on dry weight basis. Refer to the *SOP for Determination of Percent Solids* (MET-160.3). If the determination is performed by the organics preparation personnel, a portion of the sample for this determination should be weighed out at the same time as the portion used for analytical determination.

10.4. Extraction method for samples expected to contain low concentrations of organics and pesticides (<= 20 mg/Kg):

10.4.1. The following step should be performed rapidly to avoid loss of the more volatil extractables. Weigh approximately 30 g, or a weight prescribed in the determinative method, of sample into a beaker. Record the weigh to the nearest 0.1 g. Nonporous

or wet samples (gummy or clay type) that do not have a freeflowing sandy texture must be mixed with approximately 60 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added. After addition of sodium sulfate, the sample should be free flowing.

10.4.2. Add amount of surrogate standards specified in the analytical method to all samples, spikes, standards, and blanks. For the LCS and sample(s) in each analytical batch selected for matrix spiking, add the specified amount of matrix spike standard as specified in the analytical method.

10.4.3. Immediately add 100 ml of 1:1 methylene chloride:acetone (or of alternative solvent specified in the analytical method).

10.4.4. Place the bottom surface of the tip of the disrupter horn about 1/2 in. below the surface of the solvent, but above the sediment layer. Addition of a small amount of extraction solvent may be needed to ensure proper horn operation.

10.4.5. Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). Do not use microtip probe.

10.4.6. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporator flask.

10.4.7. Decant and filter extracts into a K-D apparatus using vacuum filtration and a modified funnel, covering the glass wool with sodium sulfate.

10.4.8. Repeat the extraction two or more times with two additional 100 ml (or more if needed) portions of solvent. Decant off the solvent into the K-D apparatus through the modified funnel after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample into the modified funnel and rinse with extraction solvent. Rinse 3 times with DCM.

10.4.9. Add one to two clean boiling chips to the evaporation flask, and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90-C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of

liquid reaches 10 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

10.4.10. If a solvent exchange is required, momentarily remove the Snyder column, add 15 ml of the exchange solvent and a new boiling chip, and re-attach the Snyder column. This solvent exchange should be performed in a hood with the extract near room temperature. Concentrate the extract as described in Section 7.3.8, raising the temperature of the water bath, if necessary, to maintain proper distillation. When the apparent volume again reaches 10 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

10.4.11. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 10 ml of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the nitrogen blowdown technique (section 10.3.12) or adjusted to 10.0 ml with the solvent last used.

10.4.12. Nitrogen Blowdown Technique

10.4.12.1. Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

10.4.12.2. The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). The volume of extract in the tube must be monitored during blowdown to avoid loss of more volatile analytes. Under normal operating conditions, the extract should not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

10.4.13. Bring the extract to the prescribed final volume and transfer the concentrated extract to the appropriate labeled autosampler vial or storage vial. The extracts obtained may now be analyzed for the target analytes using the appropriate determinative technique.

11. QUALITY CONTROL

11.1. Refer to the SOP for the determinative method and SOP for *Analytical Batches and Analytical Sequences* for minimum QC requirements. Project-specific batching protocols may also be required.

11.2. The QC solutions required by the method must be added as described in the analytical method. The amount and identification of QC solutions added must be documented on the benchsheet. Any reagent blanks, laboratory control samples, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

12. DATA REPORTING AND REVIEW

12.1. Preparation of all samples must be documented on a benchsheet. All information regarding the sample(s) extracted, aliquoting, QC spiking, extraction steps, etc. must be documented by the person(s) performing the extraction.

12.2. The benchsheet must be reviewed my the extraction lead, supervisor, or instrument lab analyst. The instrument lab analyst should sign-off on the benchsheet, thus accepting custody of the extracts.

13. REFERENCES

13.1. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3550B, Revision 2

13.2. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update II, September 1994, Method 3550A, Revision 1

13.3. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3500B, Revision 2

Attachment A

Benchsheets

Columbia Analytical Services, Inc.
8270C in Soil *3550B*

Serv. Req. IDs: _____ KWG #: _____

Lab Code	Client ID (as written on container)	SampleWt. (grams)	Percent Solids	Dry Weight (grams)	Xtrn. F.V. (ml)	GPC Dil.	GPC F.V.	Calc. F.V.

Comments: _____

Spike Information

Surrogate: _____ X.P. _____

Matrix Spike: _____ X.P. _____

_____ X.P. _____

Witness: _____

Extract Information

Start Date: _____ End Date: _____

Sonic Horns Tuned ☐ S-Evap Temp. _____

DCM Lot _____ Acetone Lot _____

Na_2SO_4 Lot _____ Pipette Lot _____

Extract Storage _____

Personnel and Bench Sheet Review

Started By: _____ Assisted by: _____

Completed By: _____ Assisted By: _____

Reviewed By: _____ Date: _____

Training

Yes No

Yes No

Extracts Examined

Yes No

Columbia Analytical Services, Inc.
SIM-PAH in Soil *3550B*

Serv. Req. IDs: _____ KWG #: _____

Lab Code	Client ID (as written on container)	SampleWt. (grams)	Percent Solids	Dry Weight (grams)	Xtrn. F.V. (ml)	GPC Dil.	GPC F.V.	Calc. F.V.

Comments: _____

Spike Information

Surrogate: _____ X.P. _____
Matrix Spike: _____ X.P. _____
_____ X.P. _____

Witness: _____

Extract Information

Start Date: _____ End Date: _____
Sonic Horns Tuned ☐ S-Evap Temp. _____
DCM Lot _____ Acetone Lot _____
Na_2SO_4 Lot _____ Pipette Lot _____
Extract Storage _____

Personnel and Bench Sheet Review

Started By: _____ Assisted by: _____
Completed By: _____ Assisted By: _____
Reviewed By: _____ Date: _____

Training
Yes No
Yes No

Extracts Examined
Yes No

Columbia Analytical Services, Inc.
SIM-TCL in Soil *3550B*

q. IDs: _____ KWG #: _____

Lab Code	Client ID (as written on container)	SampleWt. (grams)	Percent Solids	Dry Weight (grams)	F.V.

nts: _____

Spike Information

Surrogate: _____ X.P. _____

Matrix Spike: _____ X.P. _____

_____ X.P. _____

Witness: _____

Extract Information

Start Date: _____ End Date: _____

Sonic Horns Tuned ___ S-Evap Temp. _____

DCM Lot _____

Na_2SO_4 Lot _____

H_2SO_4 Lot _____

Extract Storage _____

Personnel and Bench Sheet Review

Started By: _____ Assisted by: _____

Completed By: _____ Assisted By: _____

Reviewed By: _____ Date: _____

Training

Yes No

Yes No

Extracts Examined

Yes No

COLUMBIA ANALYTICAL SERVICES, INC.

Work Request #:_____ **Date Extracted:**_____

Analyst:_____ **Method:**_____

8310 in Soil

Lab ID	Client ID	Sample Wt	% Solids	Dry Wt	Surr	MS	Initial Vol.	Aliq. Vol.	*True Final Vol.	C-U

*Final Volume is calculated as follows: Initial Extract Volume (ml) X Final (concentrated) Volume of the Aliquot (ml)

Note in comments Aliquot final volume Aliquot Volume (ml)

Comments:_____

_____

_____

_____

DCM Lot #_____**Acetone Lot #**_____**ACN Lot#.**_____**S-evap Temp.**_____**N-evap Temp.** _____

Soxhlet Time/Date/Initial Start_____ **Time/Date/Initial Stop**_____

Surrogate ID: _____ **Spike ID :** _____

Vial Storage:_____ **Extract Storage:**_____

Witnessed by:	Date:
Reviewed by:	Date:

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request No.: _____ Date Extracted: _____

Analyst: _____ Method: _____

Work Group #_____

Pesticides/PCBs in Soil

Lab ID	Client ID	Sample Weight	% Solids	Dry Weight	Surr	MS	Int. Vol	Solv xch	C-U

Comments:_____

_____

Sulfate Lot #_____ S-Evap Temp._____

DCM Lot #_____ Acetone Lot #_____ Hexane Lot #_____

Soxhlet Time/Date/Initial Start: _____ Time/Date/Initial Stop: _____

Surrogate ID: _____

Spike ID : _____

Spike ID : _____

Vial: _____ Extract Storage: _____ Extract Received: _____

Witnessed By:	Date:
Reviewed By:	Date:

Filename: GCBENCH/PESTPCBS/06-..J-99

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request No.: _____ Date Extracted: _____

Analyst: _____ Method: _____

Pesticides/PCBs in Soil
Low-Level Method

Lab ID	Client ID	Sample Weight	% Solids	Dry Weight	Surr	MS	Int. Vol	Solv xch	CU	Final vol

Comments: _____

_____

_____

DCM lot # _____ Acetone lot # _____ Hexane lot # _____

S-evap temp before GPC _____ after _____

Surrogate ID: _____

Spike ID : _____

Vial: _____ Extract Storage: _____

Witnessed By:	Date:
Reviewed By:	Date:

Filename: PSTPCBL2.BCH/04-09-97

APPENDIX A

Sonic Horn Tuning Procedures

Sonic Horn Tuning Procedures

Tekmar Model 501

TUNING

Tuning optimizes performance and insures maximum transfer of energy by matching the frequency of the power supply to that of the converter/probe assembly. The power supply should be tuned 1) every time a new probe or accessory is used, 2) on occasions to compensate for the frequency variation caused by cavitation erosion, 3) following 10 minutes of continuous operation and 4) when the sample temperature is significantly higher or lower than room temperature.

The piezoelectric crystal within the converter is part of the circuitry which controls the frequency at 20 kHz. Any changes in the crystal's capacitance resulting from a variation in temperature, will cause the equipment to operate in an out-of-tune condition. For reliable performance, and equipment protection, it is important that the unit be tuned after the probe temperature has had a chance to stabilize. When relocating the Ultrasonic Processor from a very cold or very hot environment, allow 30 minutes for the unit temperature to stabilize before operating. Continuous operation causes temperature elevation in the sample. This increase in temperature is transmitted through the probe to the crystal assembly. Always tune the power supply after the probe has reached operating temperature. When working with low or high temperature samples, immerse the probe in the sample for a few minutes, withdraw the probe from the sample, then tune the power supply.

> **IMPORTANT**
>
> *Tuning must be performed in air with the probe out of the sample. While tuning, do not allow the probe to contact anything.*

To tune the power supply, proceed as follows:

1. Ensure that the probe or microtip is not immersed in the sample and that it does not come in contact with anything. If a cup horn is used, make sure that the water has been drained out of it. If a flow through cell is used, make sure that the sample has been drained out of it.
2. Set PULSER to OFF.
3. Set AMPLITUDE control to "100" (to "40" when using a microtip).

> **CAUTION**
>
> *When tuning with a microtip, never allow vibration in air for more than 10 seconds. With a microtip never allow the AMPLITUDE control to be set above the microtip limit "40". Ignoring these instructions will cause the microtip to fracture.*

4. Set ON/OFF power switch to ON. The switch will illuminate.
5. Depress the TUNE switch and rotate the TUNER clockwise or counterclockwise until a minimum (not maximum) reading (usually less than 20) is obtained on the POWER MONITOR. If minimum reading cannot be obtained, the probe, cup horn, tip, microtip, extender, or accessory is loose or out of resonance, or the power supply or converter require servicing. A loose probe will usually generate a loud piercing sound.

> **NOTE**
>
> *1. The probe is tuned to vibrate at a specific frequency - 20 kHz ± 50 Hz. If the resonant frequency of the probe has changed, due to cavitation erosion or fracturing, minimum reading will not be obtained. If minimum reading cannot be obtained, check the instrument without the probe to determine which component might be defective. If proper tuning is obtained using the converter without the probe, the probe is defective and should be changed.*
>
> *2. A loose probe will usually generate a loud piercing sound.*
>
> *3. Since the amplitude required is application dependent, and subject to the volume and composition of the sample, it is recommended that the amplitude be first set at mid-range, then empirically determined and optimized while the sample is being processed.*

6. Set the AMPLITUDE control to "20" when working with a microtip, and to "50" when working with any other probe or accessory.
7. With a dual output 600 watt Ultrasonic Processor, if two converters are going to be used simultaneously, connect at this time the second converter cable to the right converter connector.

Branson Model 450

Testing the Equipment

To determine if the equipment is operating properly, proceed as follows:

a. Mount 1/2" disruptor horn (with flat tip if tapped) to converter.

b. Set Output Control to 5, Timer to HOLD and ON/OFF switch to ON.

c. Record meter reading with horn in air.

Meter Reading: _____

Horn Size: _____

d. Fill 500 ml pyrex beaker to 500 ml level with room temperature tap water.

e. Immerse horn tip halfway to 250 ml mark. Set ON/OFF switch to ON and record meter reading.

Meter Reading_____

Testing the Equipment (continued)

Perform this test and compare readings periodically, to ensure that the equipment is operating satisfactorily. A variation of 10 between the first and second readings is acceptable. If readings are not within tolerance, refer to the Trouble Analysis Section 5.

ATTACHMENT C-2
NORTHWESTERN AQUATIC SCIENCES
TEST PROTOCOLS

TEST PROTOCOL

FRESHWATER CLAM, *CORBICULA FLUMINEA*, 28-DAY BIOACCUMULATION

1. INTRODUCTION

1.1 Purpose of Study: Laboratory bioaccumulation tests provide an estimate of contaminant uptake by benthic infauna. The purpose of this study is to expose Asiatic clams (*Corbicula fluminea*) to freshwater sediments for 28 days so that they may bioaccumulate sediment-associated contaminants. After the bioaccumulation period, clams are frozen for subsequent tissue analysis.

1.2 Referenced Method: There are currently no standard methods available for testing with this species. Therefore, this protocol is based on the Portland Harbor Sediment Management Plan - Appendix G, EPA/600/R-93/183, EPA 823-B-98-004, and ASTM E-1688.

1.3 Summary of Method: A summary of test conditions for the 28-day clam bioaccumulation test is tabulated below. The 28-day bioaccumulation test with *Corbicula fluminea* is conducted at 20°C with a 12L:12D photoperiod at an illuminance of about 50-100 footcandles. Test chambers are 10-gallon aquaria containing a 5 cm layer of sediment and 5 L or more of overlying water. Generally twenty to thirty clams are used in each replicate. The number of replicates/treatment depends on the objective of the test; five replicates are recommended for routine testing. Test organisms are not fed during the test. Each chamber receives two volume additions per day of overlying water. Overlying water can be culture water, well water, surface water, site water, reconstituted water, or dechlorinated municipal water. The test endpoint is bioaccumulation.

1. Test type:	Whole-sediment bioaccumulation test with renewal of overlying water
2. Temperature:	20± 1° C
3. Light quality:	Wide-spectrum fluorescent light
4. Illumination:	About 500 to 1000 Lux
5. Photoperiod:	12L:12D
6. Test chamber:	10gal aquaria or as required
7. Sediment volume:	5cm layer, approximately 6.5L or more
8. Overlying water volume:	5 L or more
9. Renewal of overlying water:	2 volume additions/d; continuous or intermittent (e.g., one volume addition every 12 hours)
10. Age of test organisms:	Adults (~1g/clam)
11. Loading of organisms in chamber:	Approximately 20 to thirty animals
12. Number of replicate chambers/treatment:	Depends upon the objective of the test. Five replicates are recommended for routine testing.
13. Feeding:	None
14. Aeration:	None; if dissolved oxygen in overlying water drops below 40% of saturation begin aeration.
15. Overlying water:	Culture water, well water, surface water, site water, reconstituted water , or dechlorinated municipal water
16. Overlying water quality:	Hardness, alkalinity, conductivity, pH, and ammonia at the beginning and end of a test. Temperature and dissolved oxygen daily.
17. Test duration:	28 days
18. Endpoint:	Bioaccumulation
19. Test acceptability:	Test organisms must burrow into the test sediments

2. STUDY MANAGEMENT

2.1 Sponsor's Name and Address:

_____
_____
_____
_____

2.2 Sponsor's Study Monitor:

_____

2.3 Name of Testing Laboratory:
Northwestern Aquatic Sciences
3814 Yaquina Bay Road, P.O. Box 1437
Newport, OR 97365.

2.4 Test Location: _____

2.5 Laboratory's Personnel to be Assigned to the Study:
Study Director: _____
Quality Assurance Unit: _____
Aquatic Toxicologist: _____
Aquatic Toxicologist: _____

2.6 Proposed Testing Schedule: The time between sediment collection and use in testing should be kept to a minimum; therefore, bioaccumulation testing is started as soon after sample receipt as logistically possible.

2.7 Good Laboratory Practices: The test is conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations revised August 17, 1989 (40 CFR Part 792).

3. TEST MATERIAL

The test materials are freshwater sediments. The control, reference, and test sediments are placed in clean, air-tight containers. Sediments for metals bioaccumulation should be stored in the absence of air to minimize the oxidation of reduced forms. Nitrogen can be used to fill the headspace in the containers. Glass containers are recommended for sediments polluted with either metals or organics, although high-density polyethylene and PTFE containers are also acceptable. Large organisms and extraneous materials, such as bivalves or twigs, should be removed from the sediments before storing. At the laboratory the samples are stored at 4°C in the dark. The time between sediment collection and use in testing should be kept to a minimum. A negative control sediment is collected from a clean site, and should contain no or very low concentrations of the contaminant(s) of concern. In addition, a reference sediment is normally employed as a comparison station when evaluating dredged materials.

4. TEST WATER

Test water (overlying water) at NAS is normally moderately hard synthetic water at a hardness of 80-100 mg/L as $CaCO_3$ and alkalinity of 60-70 mg/L as $CaCO_3$. Dilution water is prepared from Milli-Q reagent

grade water and reagent grade chemicals. Test water may also be well water, surface water, site water or dechlorinated municipal water depending on the study needs.

5. TEST ORGANISMS

5.1 Species: clam, *Corbicula fluminea.*

5.2. Source: Commercial suppliers.

5.3 Age: adult (~1g/clam)

5.4 Acclimation and Pretest Observation: After receipt from the supplier, clams should be held in the laboratory for at least 24 hours prior to test initiation in order to assess their health and acclimate them to test conditions. Mortality during the holding period should not be excessive

6. DESCRIPTION OF TEST SYSTEM

6.1 Test Chambers and Environmental Control: Test chambers generally used in the toxicity test are 10-gallon aquaria. Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath or by placement in a temperature-controlled room. Aeration is not employed unless dissolved oxygen drops below 40% saturation. The test is conducted under an illuminance of 50-100 footcandles with a 12L:12D photoperiod.

6.2 Cleaning: All laboratory glassware, including test chambers, is cleaned as described in EPA/600/4-90/027F. New glassware and test systems are soaked 15 minutes in tap water and scrubbed with detergent (or cleaned in automatic dishwasher); rinsed twice with tap water; carefully rinsed once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases; rinsed twice with deionized water; rinsed once with acetone to remove organic compounds (using a fume hood or canopy); and rinsed three times with deionized water. Test systems and chambers are rinsed again with dilution water just before use.

7. EXPERIMENTAL DESIGN AND TEST PROCEDURES

7.1 Experimental Design: The test involves exposure of clams to test, control, and reference sediments. The sediments are placed on the bottom of the test containers and are overlain with test water. The test exposure is for 28 days. The renewal of overlying water consists of two volume additions per day, either continuous or intermittent. Each treatment consists of five replicate test containers, each containing approximately 20 to thirty organisms. Test chamber positions are completely randomized. Test organisms are randomly distributed to the test chambers. Animals are placed on the sediment surface and allowed to bury.

7.2 Setup of Test Containers: Sediments are homogenized and placed in test chambers on the day before addition of test organisms. A 5-cm layer of sediment (~6.5 L) is placed into each of five replicate aquaria. After addition of the sediment, approximately 5 L of test water is gently added to each test container in a manner to prevent resuspension. The overlying water is replaced twice daily. The test begins when clams are introduced to the test chambers. Initial water quality measurements are taken prior to the addition of test organisms.

7.3 Test Conditions: No aeration is employed unless dissoved oxygen falls below 40% saturation. The test temperature employed is 20°C (range of ± 1°C). A 12:12, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps at 50-100 footcandles. The overlying water is replaced twice daily.

7.4 Beginning the Test: The test is begun by adding the organisms to the equilibrated test containers as previously described. A five replicate zero-time sample of test animals is preserved (frozen) for analysis of initial concentrations of chemicals of concern.

7.5 Feeding: None.

7.6 Test Duration, Type and Frequency of Observations, and Methods: The duration of the bioaccumlation test is 28 days. The type and frequency of observations to be made are summarized as follows:

TYPE OF OBSERVATION	TIMES OF OBSERVATION
BIOLOGICAL DATA	
Survival	Daily; any dead animals are removed
PHYSICAL AND CHEMICAL DATA	
Hardness, alkalinity, conductivity, pH and total ammonia	Beginning and end of test in overlying water. One replicate per treatment.
Dissolved oxygen, temperature	Daily in overlying water. One replicate per treatment

Dissolved oxygen is measured using a polarographic oxygen probe calibrated according to the manufacturer's recommendations. The pH is measured using a pH probe and a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured with a calibrated mercury thermometer or telethermometer. Conductivity is measured with a conductivity meter. Hardness and alkalinity are measured using titrometric methods. Ammonia-nitrogen is measured using the salicylate colerimetric method (Clin. Chim. Acta 14:403, 1996).

7.7 Test Termination and Depuration: At test termination, animals are removed from the sediment via gently sieving test chamber contents. Any gaping animals that are unresponsive to gentle prodding should be considered dead and excluded from subsequent tissue analysis. All surviving organisms from an individual replicate should be transferred to an aquarium containing clean water for 24 hours to purge their gut contents. After this 24-hour period, animals are placed in clean containers and frozen for subsequent tissue residue analysis.

7.8 Criteria of Test Acceptance: The test results are acceptable if the test organisms burrow into the sediments; the test should be considered invalid if overt sediment avoidance is observed.

8. DATA ANALYSIS

The endpoint of the test is bioaccumulation. Surviving clams are depurated for 24 hours and then frozen for subsequent tissue analysis. Data analysis consists of calculating means and standard deviations for tissue chemical concentrations and water quality parameters. Statistical comparisons of treatment groups may be done using standard hypothesis test procedures (i.e. test for normality and homogeneity followed by parametric or non-parmetric comparison tests as appropriate).

9. REPORTING

The final report of the test results must include all of the following standard information at a minimum: name and identification of the test; the investigator and laboratory; date and time of test beginning and end; information on the test material; information on the source and quality of the overlying/test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including water quality; definition of the effect criteria and

other observations; responses, if any, in the control treatment; tabulation and statistical analysis of measured responses and a summary table of endpoints; a description of the statistical methods used; any unusual information about the test or deviations from procedures.

10. STUDY DESIGN ALTERATION

Amendments made to the protocol must be approved by the sponsor and study director and should include a description of the change, the reason for the change, the date the change took effect and the dated signatures of the study director and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

11. REFERENCED GUIDELINES

ASTM. 1997. Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates. ASTM Standard Method No. E 1688 – 97a. Am. Soc. Test. Mat., Philadelphia, PA.

Portland Harbor Sediment Management Plan. June 25, 1999. Oregon Department of Environmental Quality.

U.S. EPA. 1993. Guidance Manual: Bedded Sediment Bioaccumulation Tests: Bedded Sediment Bioaccumulation Tests. EPA/600/R-93/183.

U.S. EPA. 1998. Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Testing Manual: Inland Testing Manual. EPA 823-B-98-004.

Weber, C.I. (Ed.) 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (Fourth Edition). EPA/600/4-90/027F.

12. APPROVALS

_____ for _____
Name Date

_____ for Northwestern Aquatic Sciences
Name Date

TEST PROTOCOL

FRESHWATER OLIGOCHAETE, *LUMBRICULUS VARIEGATUS*, 28-DAY BIOACCUMULATION

1. INTRODUCTION

1.1 Purpose of Study: Laboratory bioaccumulation tests provide an estimate of contaminant uptake by benthic infauna. The purpose of this study is to expose oligochaetes (*Lumbriculus variegatus*) to freshwater sediments for 28 days so that they may bioaccumulate sediment-associated contaminants. After the bioaccumulation period, worms are frozen for subsequent tissue analysis.

1.2 Referenced Method: This protocol is based on EPA Method 100.3 (EPA/600/R-94/024) and ASTM Method E 1688-97a (ASTM 1997).

1.3 Summary of Method: A summary of test conditions for the 28-day bioaccumulation test is tabulated below. Because *L. variegatus* is quite sensitive to some contaminants, a 96-hour toxicity screening test is performed to ensure that the samples is not overly toxic prior to setting up the bioacummulation test. The screening test is conducted in 300 ml test chambers containing 100 ml of sediment and 175 ml of overlying water. The test is conducted at 23°C, 16:8 photoperiod, with twice daily renewal of overlying water. The screening test is inititated with ten adult oligochaetes per replicate and a minimum of four replicates per treatment. Animals are not fed during the test. If there is significant mortality or animals are exhibiting avoidance behavior, then bioaccumulation testing with *L. variegatus* may not be possible or appropriate. If the screening test indicates that the test sediments are not toxic and animals are not avoiding the sediment, then the bioaccumlation test is started. The 28-day bioaccumulation test with *Lumbriculus variegatus* is conducted at 23°C with a 16L:8D photoperiod at an illuminance of about 50-100 footcandles. Test chambers are 4- to 6-L aquaria containing a minimum of 1L of sediment (uniform amount across all containers) and approximately 1L or more of overlying water. Sediments are added to test chambers and overlying water exchanged twice during the 24 hours prior to test initiation. Adult oligochaetes are used. The test is stocked at densities between 1 and 5 grams wet biomass per replicate test chamber or more depending upon chemistry requirements as long as the minimum ratio of sediment TOC to organism dry weight of 50:1 is not violated. There are five replicates per treatment. Test organisms are not fed during the test. Aeration is not used unless dissolved oxygen falls below 40% saturation. Each chamber receives two volume additions per day of overlying water. Overlying water can be culture water, well water, surface water, site water, reconstituted water, or dechlorinated municipal water. The test endpoint is bioaccumulation.

1. Test type:	Whole-sediment bioaccumulation test with renewal of overlying water
2. Temperature:	23± 1° C
3. Light quality:	Wide-spectrum fluorescent light
4. Illumination:	About 500 to 1000 Lux
5. Photoperiod:	16L:8D
6. Test chamber:	4- to 6-L aquaria
7. Sediment volume:	1 L or more depending upon TOC
8. Overlying water volume:	1 L or more depending upon TOC
9. Renewal of overlying water:	2 volume additions/day; continuous or intermittent (e.g., one volume addition every 12 hours)
10. Age of test organisms:	Adults
11. Loading of organisms in chamber:	Ratio of total organic carbon in sediment to organism dry weight should be no less than 50:1. Minimum of 1g/replicate. Preferably 5g/replicate.
12. Number of replicate chambers/treatment:	Depends upon the objective of the test. Five replicates are recommended for routine testing.
13. Feeding:	None

14. Aeration:	None, unless dissolved oxygen in overlying water drops below 40% of saturation.
15. Overlying water:	Culture water, well water, surface water, site water, or reconstituted water, or dechlorinated municipal water.
16. Overlying water quality:	Hardness, alkalinity, conductivity, pH, and ammonia at the beginning and end of a test. Temperature and dissolved oxygen daily.
17. Test duration:	28 days
18. Endpoint:	Bioaccumulation
19. Test acceptability:	Test organisms must burrow into the test sediments.

2. STUDY MANAGEMENT

2.1 Sponsor's Name and Address:

_____
_____
_____
_____

2.2 Sponsor's Study Monitor:

_____

2.3 Name of Testing Laboratory:
Northwestern Aquatic Sciences
3814 Yaquina Bay Road, P.O. Box 1437
Newport, OR 97365.

2.4 Test Location: _____

2.5 Laboratory's Personnel to be Assigned to the Study:
Study Director: _____
Quality Assurance Unit: _____
Aquatic Toxicologist: _____
Aquatic Toxicologist: _____

2.6 Proposed Testing Schedule: The time between sediment collection and use in testing should be kept to a minimum; therefore, bioaccumulation testing is started as soon after sample receipt as logistically possible.

2.7 Good Laboratory Practices: The test is conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations revised August 17, 1989 (40 CFR Part 792).

3. TEST MATERIAL

The test materials are freshwater sediments. The control, reference, and test sediments are placed in clean, air-tight containers. Sediments for metals bioaccumulation should be stored in the absence of air to minimize the oxidation of reduced forms. Nitrogen can be used to fill the headspace in the containers. Glass containers are recommended for sediments polluted with either metals or organics, although high-density polyethylene and PTFE containers are also acceptable. Large organisms and extraneous materials, such as bivalves or twigs, should be removed from the sediments before storing. At the laboratory the samples are stored at 4°C in the dark. The time between sediment collection and use in testing should be kept to a minimum. A negative control sediment is collected from a clean site, and should contain no or

very low concentrations of the contaminant(s) of concern. In addition, a reference sediment is normally employed as a comparison station when evaluating dredged materials

4. TEST WATER

Test water (overlying water) at NAS is normally moderately hard synthetic water at a hardness of 80-100 mg/L as $CaCO_3$ and alkalinity of 60-70 mg/L as $CaCO_3$. Dilution water is prepared from Milli-Q reagent grade water and reagent grade chemicals. Test water may also be well water, surface water, site water or dechlorinated municipal water depending on the study needs.

5. TEST ORGANISMS

5.1 Species: oligochaete, *Lumbriculus variegatus.*

5.2. Source: Commercial suppliers or laboratory cultures

5.3 Age: Adult

5.4 Acclimation and Pretest Observation: After receipt from the supplier, worms should be held in the laboratory for at least 24 hours prior to test initiation in order to assess their health and acclimate them to test conditions. Mortality during the holding period should not be excessive.

6. DESCRIPTION OF TEST SYSTEM

6.1 Test Chambers and Environmental Control: Test chambers generally used in the toxicity test are 4- to 6-L aquaria. Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath or by placement in a temperature-controlled room. Aeration is not empolyed unless dissolved oxygen drops below 40% saturation. The test is conducted under an illuminance of 50-100 footcandles with a 16L:8D photoperiod.

6.2 Cleaning: All laboratory glassware, including test chambers, is cleaned as described in EPA/600/4-90/027F. New glassware and test systems are soaked 15 minutes in tap water and scrubbed with detergent (or cleaned in automatic dishwasher); rinsed twice with tap water; carefully rinsed once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases; rinsed twice with deionized water; rinsed once with acetone to remove organic compounds (using a fume hood or canopy); and rinsed three times with deionized water. Test systems and chambers are rinsed again with dilution water just before use.

7. EXPERIMENTAL DESIGN AND TEST PROCEDURES

7.1 Experimental Design: The test involves exposure of worms to test, control, and reference sediments. The sediments are placed on the bottom of the test containers and are overlain with test water. The test exposure is for 28 days. The renewal of overlying water consists of two volume additions per day, either continuous or intermittent. Each treatment consists of five replicate test containers, each containing enough organisms to provide approximately 1- to 5-g wet weight. The animals are added to each replicate at about 1.33 times the target stocking weight (the additional 33% accounts for the excess weight from water in the nonblotted oligochaetes). Test chamber positions are completely randomized. Test organisms are randomly distributed to the test chambers. Animals are placed on the sediment surface and allowed to bury.

7.2 Setup of Test Containers: Sediments are homogenized and placed in test chambers on the day before addition of test organisms. At least 1-L of sediment is placed into each of five replicate aquaria. After addition of the sediment, 1 L or more of test water is gently added to each test container in a manner to prevent resuspension. The overlying water is replaced twice daily. The test begins when worms are introduced to the test chambers. Initial water quality measurements are taken prior to the addition of test organisms.

7.3 Test Conditions: No aeration is employed unless dissoved oxygen falls below 40% saturation. The test temperature employed is 23°C (range of ± 1°C). A 16:8, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps at 50-100 footcandles. The overlying water is replaced twice daily.

7.4 Beginning the Test: The test is begun by adding the organisms to the equilibrated test containers as previously described. A five replicate zero-time sample of test animals is preserved (frozen) for analysis of initial concentrations of chemicals of concern.

7.5 Feeding: None.

7.6 Test Duration, Type and Frequency of Observations, and Methods: The duration of the bioaccumlation test is 28 days. The type and frequency of observations to be made are summarized as follows:

TYPE OF OBSERVATION	TIMES OF OBSERVATION
BIOLOGICAL DATA	
Survival and observations on behavior	Daily; any dead animals are removed
PHYSICAL AND CHEMICAL DATA	
Hardness, alkalinity, conductivity, pH and total ammonia	Beginning and end of test in overlying water. One replicate per treatment.
Dissolved oxygen, temperature	Daily in overlying water. One replicate per treatment

Dissolved oxygen is measured using a polarographic oxygen probe calibrated according to the manufacturer's recommendations. The pH is measured using a pH probe and a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured with a calibrated mercury thermometer or telethermometer. Conductivity is measured with a conductivity meter. Hardness and alkalinity are measured using titrometric methods. Ammonia-nitrogen is measured using the salicylate colerimetric method (Clin. Chim. Acta 14:403, 1996).

7.7 Test Termination and Depuration: At test termination, animals are removed from the sediment via gently sieving test chamber contents through a fine-mesh sieve sufficiently small to retain the oligochaetes (500 µm mesh). Immobile organisms should be considered dead. Live oligochaetes from an individual replicate should be transferred to a 1-L beaker containing overlying water without sediment for 24 hours to depurate, or eliminate gut contents. Aeration may be required if dissolved oxygen falls below 40%. Each sample is then weighed, placed in a clean container, and frozen for later tissue residue analysis.

7.8 Criteria of Test Acceptance: The test results are acceptable if the test organisms burrow into the sediments; the test should be considered invalid if overt sediment avoidance is observed.

8. DATA ANALYSIS

The endpoint of the test is bioaccumulation. Surviving worms are depurated for 24 hours and then frozen for subsequent tissue analysis. Data analysis consists of calculating means and standard deviations for tissue chemical concentrations and water quality parameters. Statistical comparisons of treatment groups

may be done using standard hypothesis test procedures (i.e. test for normality and homogeneity followed by parametric or non-parmetric comparison tests as appropriate).

9. REPORTING

The final report of the test results must include all of the following standard information at a minimum: name and identification of the test; the investigator and laboratory; date and time of test beginning and end; information on the test material; information on the source and quality of the overlying/test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including water quality; definition of the effect criteria and other observations; responses, if any, in the control treatment; tabulation and statistical analysis of measured responses and a summary table of endpoints; a description of the statistical methods used; any unusual information about the test or deviations from procedures; results of the initial screening toxicity test.

10. STUDY DESIGN ALTERATION

Amendments made to the protocol must be approved by the sponsor and study director and should include a description of the change, the reason for the change, the date the change took effect and the dated signatures of the study director and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

11. REFERENCED GUIDELINES

ASTM. 1997. Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates. ASTM Standard Method No. E 1688 – 97a. Am. Soc. Test. Mat., Philadelphia, PA.

Portland Harbor Sediment Management Plan. June 25, 1999. Oregon Department of Environmental Qualtiy.

U.S. EPA. 1994. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA/600/R-94/024.

U.S. EPA. 1998. Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Testing Manual: Inland Testing Manual. EPA 823-B-98-004.

Weber, C.I. (Ed.) 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (Fourth Edition). EPA/600/4-90/027F.

12. APPROVALS

_____ for _____
Name Date

_____ for Northwestern Aquatic Sciences
Name Date

APPENDIX D – HAHN & ASSOCIATES HEALTH AND SAFETY PLAN

SITE HEALTH AND SAFETY PLAN
PORTLAND SHIPYARD
REMEDIAL INVESTIGATION

Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

December 20, 1999

Prepared by:

Hahn and Associates, Inc.
Portland, Oregon

HAI Project No. 4800

TABLE OF CONTENTS

ATTACHMENTS

SITE HEALTH AND SAFETY PLAN

Portland Ship Yard
5555 North Channel Avenue
Portland, Oregon

December 20, 1999

1.0 INTRODUCTION

This site-specific Site Health and Safety Plan (HASP) has been developed as required by the Occupational Safety and Health Administration (OSHA) according to the Code of Federal Regulations (CFR) 29 CFR 1910.120. The Site Health and Safety Plan includes discussion under the following section headings: Site Description; Organization and Coordination; Site Control; Hazard Evaluation; Personal Protective Equipment; Communication Procedures; Decontamination Procedures; and Site Safety and Health.

This Site Health and Safety Plan applies to members of the Hahn and Associates, Inc. (HAI) field staff and HAI subcontractors. The Plan is not intended for use by other consultants or contractors working at the site. In addition, this HASP applies only to upland areas of the Portland Ship Yard (PSY) site (the "Site").

Based on the results of previous investigatory activities performed at the site, as well as available information regarding site operations, Level D protection is recommended for all work activities. However, the level of protection may be upgraded to Level C if applicable conditions within Section 6.0 of this plan are identified.

2.0 SITE DESCRIPTION

2.1 Site Location

The PSY is located at 5550 North Channel Avenue, Portland, Oregon, at the northwest end of Swan Island. The site is bordered on three sides by water bodies including the Willamette River and Swan Island Lagoon, and to the southeast by North Channel Avenue and industrial properties.

2.2 Site Features

A variety of structures are present at the Site. In addition, an above-ground storage tank (AST) farm and three dry docks are located at the Site. The topography at the Site is generally level at an elevation of approximately 40 feet above mean sea level (msl). The property is generally asphalt or concrete covered with little or no vegetation, with the exception of the Channel Fabrication Site that is generally unpaved.

2.3 Site History and Contaminants of Concern

Operations at the PSY from 1942 to present consist of ship repair and maintenance. Based on a May 26, 1999, Oregon Department of Environmental Quality (DEQ) memorandum, the contaminants of potential concern at the Site include total petroleum hydrocarbons (TPH) and hydrocarbon constituents, including benzene, toluene, ethylbenzene, and xylene (BTEX) and polynuclear aromatic hydrocarbons (PAHs), volatile organic compounds (VOCs), polychlorinated biphenyls (PCBs), tributyl tin (TBT), and metals.

3.0 ORGANIZATION AND COORDINATION

4.1 Site Personnel/Team Organization

Team Member	Responsibility	Work Zone(s)
Guy Tanz	HAI Project Supervisor, Health and Safety Officer	All
Jay Greifer	HAI Field Team Leader, Site Safety Coordinator	All
Field Team	Conduct field activities	All

4.2 Work Team Job Functions

Health and Safety Officer	Administer HAI's HASP and ensure HASP, accident reports
Site Safety Coordinator	Carry out all aspects of HASP, ensure all HAI employees and subcontractors adhere to HASP
Field Team Leader	Supervise, obtain samples, air monitoring, logging
Field Team Member(s)	Drilling, surveying, excavating

All personnel arriving or departing the site should log in and out with the Site Safety Coordinator. All activities on site must be cleared through the Project Team Leader.

4.0 SITE CONTROL

1) Conduct a daily site safety meeting to discuss each day's planned activities and review the HASP, particularly communications. Additional topics should include:
   - Location of nearest telephone and post the emergency telephone numbers.
   - Location of nearest hospital and post the location.
   - Designation and location of emergency vehicle and location of operating keys.
   - Days weather report and weather conditions, in particular wind direction.
   - A discussion of any unexpected conditions/hazards (windy conditions etc.).
   - Determine location of support zone and decontamination.
   - A discussion of work zones and any necessary modifications to levels of protection required.
   - Review emergency site egress point(s).

2) Maintain access to the site and exclusion zones during the project duration. Exclusion zones should be developed and maintained during the work activities.

3) Team members don specified level of protection prior to entering any temporary exclusion zone. The entire decontamination process will be conducted prior to exiting.

5.0 HAZARD EVALUATION

There are two general forms of hazards expected at the site that include: 1) physical hazards; and 2) chemical hazards.

5.1 Physical Hazards

Physical hazards generally include heavy equipment, vehicles, utilities (overhead and underground), and other hazards such as slip, trip, and fall. It is expected that all of these hazards will likely be present at the site. Personnel at the site should be remain aware of such hazards.

5.2 Chemical Hazards

Site specific hazardous substances have been identified to be present in the soils and may be present in the groundwater at the site. Based on the DEQ memorandum, the contaminants of potential concern for the site include:

- TPH
- BTEX
- VOCs
- PAHs
- PCBs
- Various metals

Personnel may be exposed to these constituents during on-site activities.

In general, primary exposure routes of the identified hazards include dermal contact, incidental oral ingestion of contaminant-laden soils and/or dust and inhalation of vapors or dust. Material safety data sheets (MSDS) for primary contaminants of concern for the site are included in Attachment A. For a summary of symptoms via exposure routes for primary constituents refer to the MSDS sheets.

5.3 Hazard Zone Delineation

Physical hazards are of concern across the entire site and chemical hazards are of concern at the specific areas to be investigated. As such, the entire site is considered to warrant Level D personal protective equipment (PPE) while upgrade to Level C may be warranted under certain conditions at areas of investigation as described in Section 6.0.

6.0 PERSONAL PROTECTIVE EQUIPMENT

6.1 Basic Equipment and Levels of Protection

Level D: Work coveralls or tyvek suits, steel toe/shank boots, safety glasses, and hard hat

Level C: Level D plus dust resistant Tyvek coveralls, disposable nitrile or vinyl gloves, half-face respirator with appropriate cartridge

HAI does not conduct work in Level A or Level B environments.

Level D PPE can be used when the atmosphere contains no known hazard; oxygen concentrations are not less than 19.5%; and work functions preclude splashes, immersion, or the potential for unexpected inhalation of a contact with hazardous levels of chemicals.

Level C PPE can be used when oxygen concentrations are not less than 19.5 %; atmospheric contaminants, liquid splashes, or other direct contact will not adversely affect any exposed skin; types of air contaminants have been identified, concentrations measured, and a cartridge or canister is available that can remove the contaminant; atmospheric contaminant concentrations do not exceed immediately dangerous to life and health (IDLH) levels; and job functions do not require self-contained breathing apparatus.

Modification of the Level C PPE equipment will be the decision of the Site Safety Coordinator.

6.2 Applicability of Basic PPE and Safety Equipment

Zone	Level of Protection/PPE	Safety Equipment
All areas	Level D	Emergency vehicle first aid kit, blanket, fire extinguisher, mobile telephone, drinking water
Exclusion Zone, Contamination-Reduction Zone, Decontamination Line	Level C	As above, decontamination water, eyewash, tape, extra gloves

Upgrade to Level C PPE will take place when either of the following conditions occur:

- Ambient air quality, measured by a PID exceeds a threshold level of 10 ppm [time weighted average (TWA) for benzene for some sub-segments of industry] for more than two consecutive readings spaced approximately 60 minutes apart in areas where VOCs have not been detected, and benzene and/or vinyl chloride levels exceed a threshold of 1 ppm via detector tubes. The frequency of air monitoring will be increased to 15 minute intervals in areas where VOCs have been detected or are thought to have been encountered.

- Visible air-borne dust is present in work areas of known or suspected metals or SVOC contamination.

7.0 COMMUNICATION PROCEDURES

1) Daily Procedures

- Prior to beginning daily field activities, a tailgate meeting will be held in the Support/Clean zone to review project status, work objectives, zone delineation, present site conditions, levels of protection, individual team member responsibilities, access and egress points, and decontamination procedures.

2) Field Communication Procedures

- Field activities will be directed using oral communications with appropriate hand signals to be used.

- A minimum of one cellular phone will be on-site during all field activities and will be designated as the field phone for use by team members.

8.0 DECONTAMINATION PROCEDURES

1) Decontamination Procedures (for personnel, equipment, meters, samples, etc.):

- Personnel: If disposable booties are worn, these should be discarded; neoprene boots should be washed with potable water and trisodium phosphate (TSP) or Alconox solution, followed by two separate potable water rinses. Remove tyveks and discard. Remove gloves and discard.

- Equipment: All drilling equipment will be steam-cleaned between drilling locations to prevent cross-contamination between borings. All soil sampling equipment will be decontaminated after each sample by using a detergent solution wash, followed by two potable water rinses.

- Emergency decontamination will include all of the specified steps to the extent practicable.

2) Material Disposal Methods

- Contaminated Articles: Disposable personnel protective equipment and sampling equipment will be placed in plastic bags and disposed as solid waste. Bags of soiled equipment will not be accessible to the public prior to disposal (to eliminate scavenging of these articles).

- All soil cuttings from drilled soil borings will be placed in 55-gallon drums and characterized to determine appropriate disposal.

- Decontamination water will be collected in 55-gallon drums and characterized to determine appropriate disposal.

Note: It is the responsibility of the Site Safety Coordinator to make sure that all pieces of equipment coming off site are properly decontaminated according to the procedures outlined above.

9.0 SITE SAFETY AND HEALTH

9.1 Designated Site Health and Safety Officers

HAI Site Safety Officer
Guy Tanz, Sr. Project Mgr.
(503) 796-0717
gtanz@hahnasoc.com

HAI Health and Safety Officer
Rob Ede, Sr. Project Mgr.
(503) 796-0717
rede@hahnasoc.com

HAI Mailing Address
434 NW 6th Avenue, Su. 203
Portland, Oregon 97209

HAI Corporate Management
Mr. Roger Brown, Principal
(503) 796-0717
rbrown@hahnasoc.com

HAI Site Safety Coordinater
Mr. Jason Greifer, Scientist
(b) (6) (mobile phone)
jgreifer@hahnasoc.com

9.2 Emergency Medical Care

Entity	Phone or Address
Police	911
Fire	911
Paramedic	911
Hospital	Legacy Emanuel Hosipital and Health Center 2801 North Gantenbein Avenue Portland, OR 97210 (503) 413-2200 Contact: Emergency
Poison Control Center	Oregon Poison Center (503) 494-8968 Contact: Emergency

9.3 Environmental Monitoring

The breathing zone and ambient air quality of work areas will be monitored for organic vapors in areas where such contaminants are suspected. The work breathing zone in these areas will be continuously monitored utilizing a MicroTIP equipped with a photoionization detector (PID) with a 10.6 electron volt (eV) lamp. If any organic vapor levels are detected above a threshold level of 10 ppm for more than two consecutive readings, spaced approximately 60 minutes apart, then work will stop, the cause for the elevated readings evaluated, and PPE may be upgraded to Level C. At the time of evaluation, benzene and/or vinyl chloride levels will be analyzed via detector tube, and if a threshold of 1 ppm is exceeded PPE will be upgraded to Level C. The frequency of air monitoring will be increased to 15 minute intervals in areas where VOCs have been detected or are thought to have been encountered.

9.4 Work Limitations

1) When in Level C, schedule beverage/rest breaks of a minimum of 15 minutes in a shaded area every 2 hours when ambient temperature exceeds 75° F. When ambient temperatures exceed 80° F, provide a beverage/rest breaks of a minimum of 15 minutes in a shaded area every 1 1/2 hours.

2) Work will be performed only during daylight hours unless adequate lighting is provided.

3) No eating, drinking, or smoking within contamination zones.

4) No facial hair is acceptable that would interfere with respirator fit.

5) No contact lenses on site.

6) Buddy system at all times in contamination zone (visual/voice contact).

9.5 Emergency Procedures

1) Basic Procedures

Stabilize injured person and remove from contamination zone, following the decontamination procedures as much as practicable

Notify the Site Safety Coordinator

The Site Safety Coordinator will direct activities during a medical emergency.

Initiate first aid and immediately get medical attention for the injured party.

Depending upon the type and severity of the injury, call the appropriate emergency response agency.

Notify the health and safety officer and HAI Corporate Management.

Prepare an incident report. The HAI Health and Safety Officer is responsible for ensuring its preparation and submittal to the health and safety officer and HAI Corporate Management within 48 hours

2) Site-specific Instructions

Acute exposure to hazardous materials:

Dermal: Wash exposed skin areas with clean water and reassess personnel protective equipment to include adequate splash protection. Call physician when exposure is suspected.

Eye: Wash eye for 15 minutes with clean water and get immediate medical attention. Do not delay eyewash for any reason.

Inhalation: Move exposed person to fresh air at once. If breathing has stopped, perform mouth-to-mouth resuscitation. Keep the affected person warm and at rest. Get medical attention as soon as possible.

3) Directions to Hospital

From the site, go south on North Channel Avenue; turn left on North Going Street, turn right on North Greeley Avenue; turn left on North Russell, turn left on North Gantenbein Avenue, Legacy Emmanuel Hospital is located at 2801 North Gantenbein Avenue. A map from the site to the hospital is in Attachment C.

10.0 PLAN APPROVAL

This Site Health and Safety Plan has been written for the use of HAI, its employees, and subcontractors. HAI claims no responsibility for its use by others. The plan is written for the specific site conditions, purposes, dates, and personnel specified and must be amended if these conditions change.

Plan Prepared By: _____ Date: _____

Plan Reviewed by: _____ Date: _____

Distribution of plan:
HAI Project Manager/Site Safety Coordinator
HAI Health and Safety Officer

11.0 SIGNATURES

All HAI site personnel have read the above plan, are familiar with its provisions, and will sign below prior to initiating work at the site each day.

NAME	SIGNATURE	DATE	COMPANY

12.0 GLOSSARY OF ABBREVIATIONS

CFR	Code of Federal Regulations
HAI	Hahn and Associates, Inc.
IDLH	Immediately Dangerous to Life or Health
MSDS	material safety data sheets
OSHA	Occupational Safety and Health Administration
PPE	personal protective equipment
RCRA	Resource Conservation and Recovery Act
TSP	trisodium phosphate
TWA	8-hour Time Weighted Average

ATTACHMENT D-1

Material Data Safety Sheets

BELL FUELS -- LEAD-FREE GASOLINE; NO-LEAD GASOLINE - GASOLINE,AUTOMOTIVE
MATERIAL SAFETY DATA SHEET
NSN: 9130001487103
Manufacturer's CAGE: 8P539
[illegible] No. Indicator: A
[illegible]umber/Trade Name: LEAD-FREE GASOLINE; NO-LEAD GASOLINE

General Information

Item Name: GASOLINE,AUTOMOTIVE
Company's Name: BELL FUELS, INC
Company's Street: 4116 WEST PATERSON AVE
Company's City: CHICAGO
Company's State: IL
Company's Country: US
Company's Zip Code: 60646
Company's Emerg Ph #: 312-286-0200
Company's Info Ph #: 312-286-0200
Record No. For Safety Entry: 110
Tot Safety Entries This Stk#: 119
Status: SP
Date MSDS Prepared: 23FEB90
Safety Data Review Date: 21OCT94
Supply Item Manager: KY
MSDS Serial Number: BVGZN
Specification Number: VV-G-1690A
Spec Type, Grade, Class: CL A,B,C,D,E GR REG
Hazard Characteristic Code: F2
Unit Of Issue: GL
Unit Of Issue Container Qty: BULK
Type Of Container: BULK

Ingredients/Identity Information

Proprietary: NO
[illegible]ient: HYDROCARBONS, AROMATIC
[illegible]ent Sequence Number: 01
[illegible]nt: 15-35
NIOSH (RTECS) Number: 1008732HA
OSHA PEL: NOT ESTABLISHED
ACGIH TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: SATURATED HYDROCARBONS
Ingredient Sequence Number: 02
Percent: 60-75
NIOSH (RTECS) Number: 1006886SH
OSHA PEL: NOT ESTABLISHED
ACGIH TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: UNSATURATED HYDROCARBONS
Ingredient Sequence Number: 03
Percent: 1-15
NIOSH (RTECS) Number: 1006887UH
OSHA PEL: NOT ESTABLISHED
ACGIH TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: DYE AND OTHER ADDITIVES
Ingredient Sequence Number: 04
Percent: 0.02
NIOSH (RTECS) Number: 1003746AD
[illegible]PEL: NOT ESTABLISHED
[illegible]. TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: BLUE OR CLEAR, TYPICAL HYDROCARBON ODOR.
Boiling Point: 90.0F,32.2C
Vapor Pressure (MM Hg/70 F): 414 @100C
Vapor Density (Air=1): 3-4
Specific Gravity: 0.71-0.77
Solubility In Water: NEGLIGIBLE.

Fire and Explosion Hazard Data

Flash Point: -50F,-46C
Flash Point Method: TCC
Lower Explosive Limit: 1.3
Upper Explosive Limit: 6
Extinguishing Media: ANY UL APPROVED CLASS B MEDIA SUCH AS FOAM, CARBON DIOXIDE, DRY CHEMICAL.
Special Fire Fighting Proc: NONE SPECIFIED BY MFG; HOWEVER USE APPROPRIATE PROTECTIVE EQPMT INCLUDING SELF-CONTAINED BREATHING APPARATUS.
Unusual Fire And Expl Hazrds: NONE SPECIFIED BY MFG; HOWEVER MATL IS HEAVIER THAN AIR AND WILL TRAVEL LONG DISTANCES & FLASHBACK. EXPLOSIVE MIXTURE FORMS W/GASOLINE & AIR.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MFG; HOWEVER AVOID OPEN FLAMES/HEAT/SPARKS/OTHER IGNITION SOURCES.
Materials To Avoid: OXIDIZERS.
Hazardous Decomp Products: NONE SPECIFIED BY MFG.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT RELEVANT.

Health Hazard Data

LD50-LC50 Mixture: UNKNOWN
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Haz Acute And Chronic: ACUTE:EYE:IRRIT @ HIGH VAP LEVELS OR DIRECT CONTACT W/FLUID. SKIN:IRRIT ON PROLONG CONTACT W/LIQ, DERM RESULTING FROM DEFATTING NATURE OF LIQ. SYSTEMATIC:CNS EFFECTS (NARCOSIS) @ HIGH VAP LEVELS; MUC MEMBRANE IRRIT, PNEUMONIA. INGEST:GASTROINTESTINAL DISTRUBANCES. CHRONIC:PERIPERAL NERVOUS SY EFFECTS, BLOOD ALTERATIONS
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: PER MSDS:NONE STATED; HOWEVER CONTAINS GASOLINE WHICH IS CONSIDERED BY IARC TO BE POTENTIAL CARCINOGEN.
Signs/Symptoms Of Overexp: EYE & SKIN IRRITATION. DERMATITIS. NARCOSIS. GI DISTURBANCES:NAUSEA, DIARRHEA, STOMACH PAINS.
Med Cond Aggravated By Exp: NONE SPECIFIED BY MFG.
THOROUGHLY WASH AREA W/SOAP & WATER. INHAL:REMOVE FROM CONTAMINATED AREA. ADMINISTER ARTIFICIAL RESP IF NECESSARY. CALL PHYSICIAN. INGEST:GIVE A VEGETABLE OIL TO RETARD ABSORPTION. DO NOT INDUCE VOMITING. CALL PHYSICIAN. FATAL DOSE ADULT HUMAN APPROX 350G, CHILD APPROX 10-13G.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: KEEP PUBLIC AWAY. SHUT OFF SOURCE W/O RISK. ADVISE POLICE & NAT RESP CENTER 800-424-8802 IF SUBSTANCE HAS ENTERED A WATER COURSE OR SEWER. CONTAIN LIQ W/EARTH, SAND. RECOVER FREE LIQ BY PPUMPING OR W/SUITABLE ABSORBENT.
Neutralizing Agent: NONE SPECIFIED BY MFG.
Waste Disposal Method: UNDER MANY SPILL SITUATIONS LIQ CAN BE RECOVERED & RECLAIMED. WHERE SOLID ABSORBENTS ARE USED THEY SHOULD BE INCINERATED PER APPLICABLE STATE & LOCAL REGULATIONS.
Precautions-Handling/Storing: USE APPROPRIATE GROUNDING-DISPENSING PROCEDURES. STORE IN RELATIVELY COOL PLACE. DO NOT EXPOSE TO HEAT, OPEN FLAMES OR OXIDANTS.
Other Precautions: NONE SPECIFIED BY MFG.

Control Measures

Respiratory Protection: FOR EXPOSURES IN EXCESS OF EXPOSURE LIMITS

CHEMICAL CARTRIDGE RESPIRATOR OR AIR SUPPLIED EQUIPMENT.
Ventilation: LOCAL EXHAUST REQUIRED & EXPLOSION PROOF EQUIPMENT.
Protective Gloves: IMPERMEABLE GLOVES.
Eye Protection: NONE SPECIFIED HOWEVER SAF GLASSES/GOGG
Other Protective Equipment: NONE SPEICFIED BY MFG.
Work Hygienic Practices: WASH HANDS AFTER HANDLING & PRIOR TO EAT/DRINK/ SMOKE/USE OF TOILET FACILITIES. FOLLOW GOOD WORK HYGIENE PRACTICES.

Transportation Data

Trans Data Review Date: 94294
DOT PSN Code: GTN
DOT Proper Shipping Name: GASOLINE
DOT Class: 3
DOT ID Number: UN1203
DOT Pack Group: II
DOT Label: FLAMMABLE LIQUID
IMO PSN Code: HRV
IMO Proper Shipping Name: GASOLINE
IMO Regulations Page Number: 3141
IMO UN Number: 1203
IMO UN Class: 3.1
IMO Subsidiary Risk Label: -
IATA PSN Code: MUC
IATA UN ID Number: 1203
IATA Proper Shipping Name: GASOLINE
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: MUC
AFI Prop. Shipping Name: GASOLINE
AFI Class: 3
AFI ID Number: UN1203
AFI Pack Group: II
AFI Basic Pac Ref: 7-7

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 21OCT94
Label Status: F
Common Name: LEAD-FREE GASOLINE; NO-LEAD GASOLINE
Signal Word: DANGER!
Acute Health Hazard-Moderate: X
Contact Hazard-Moderate: X
Fire Hazard-Severe: X
Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE:EYE:IRRIT @ HIGH VAP LEVELS OR DIRECT CONTACT W/FLUID. SKIN:IRRIT ON PROLONG CONTACT W/LIQ, DERM RESULTING FROM DEFATTING NATURE OF LIQ. SYSTEMATIC:CNS EFFECTS (NARCOSIS) @ HIGH VAP LEVELS; MUC MEMBRANE IRRIT, PNEUMONIA. INGEST:GASTROINTESTINAL DISTRUBANCES. CHRONIC:PERIPERAL NERVOUS SYS EFFECTS, BLOOD ALTERATIONS. 1STAID:EYE:FLUSH FOR @ LEAST 15MINS W/WATER. SKIN:THOROUGHLY WASH AREA W/ SOAP & WATER. INHAL:REMOVE FROM CONTAMINATED AREA. ADMINISTER ARTIFICIAL RESP IF NECESSARY. CALL PHYSICIAN. INGEST:GIVE A VEGETABLE OIL TO RETARD ABSORPTION. DO NOT INDUCE VOMITING. CALL PHYSICIAN. FATAL DOSE ADULT HUMAN APPROX 350G, CHILD APPROX 10-13G.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: BELL FUELS, INC
Label Street: 4116 WEST PATERSON AVE
Label City: CHICAGO
Label State: IL
Label Zip Code: 60646
Label Country: US
Label Emergency Number: 312-286-0200

CONOCO – DIESEL FUEL NO. 2 - DIESEL FUEL
MATERIAL SAFETY DATA SHEET
NSN: 9140002865294
Manufacturer's CAGE: 15445
Part No. Indicator: A
[illegible] Number/Trade Name: DIESEL FUEL NO. 2

General Information

Item Name: DIESEL FUEL
Company's Name: CONOCO INC
Company's Street: 600 N DAIRY ASHFORD RD RM 3012
Company's P. O. Box: 4784
Company's City: HOUSTON
Company's State: TX
Company's Country: US
Company's Zip Code: 77210-4784
Company's Emerg Ph #: 713-293-5550/800-424-9300
Company's Info Ph #: 713-293-5550
Distributor/Vendor # 1: CONOCO INTERNATIONAL INC
Distributor/Vendor # 1 Cage: 5R396
Record No. For Safety Entry: 037
Tot Safety Entries This Stk#: 112
Status: SMU
Date MSDS Prepared: 14AUG91
Safety Data Review Date: 24JUN92
Supply Item Manager: KY
MSDS Serial Number: BMZTN
Specification Number: VV-F-800
Spec Type, Grade, Class: GRADE DF-2
Hazard Characteristic Code: F4
Unit Of Issue: GL
Unit Of Issue Container Qty: BULK
Type Of Container: BULK
Net Unit Weight: UNKNOWN

Ingredients/Identity Information

[illegible]etary: NO
Ingredient: PETROLEUM MID-DISTILLATE (DIESEL MARINE FUEL)
Ingredient Sequence Number: 01
Percent: 100 %
NIOSH (RTECS) Number: 1004302PE
CAS Number: 68476-34-6
OSHA PEL: 5 MG/M3 AS OIL MIST
ACGIH TLV: 5 MG/M3 AS OIL MIST
Other Recommended Limit: NONE SPECIFIED

Proprietary: NO
Ingredient: NAPHTHALENE (SARA III)
Ingredient Sequence Number: 02
Percent: 3%
NIOSH (RTECS) Number: QJ0525000
CAS Number: 91-20-3
OSHA PEL: 10 PPM/15 STEL
ACGIH TLV: 10 PPM/15 STEL; 9293
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: CLEAR OR LIGHT YELLOW LIQUID, AROMATIC ODOR
Boiling Point: 350 - 680F
Melting Point: NOT GIVEN
Vapor Pressure (MM Hg/70 F): 1 MMHG
Vapor Density (Air=1): > 1
Specific Gravity: 0.85 - 0.93
Decomposition Temperature: NOT GIVEN
[illegible] tion Rate And Ref: NIL
[illegible] y In Water: INSOLUBLE
Per[illegible]t Volatiles By Volume: NIL
Corrosion Rate (IPY): UNKNOWN
Autoignition Temperature: 536F

Fire and Explosion Hazard Data

Flash Point: 130F,54C
Flash Point Method: TCC
Lower Explosive Limit: 0.4 %
Upper Explosive Limit: 6 %
Extinguishing Media: WATER SPRAY, FOAM, DRY CHEMICAL CAARBON DIOXIDE
Special Fire Fighting Proc: USE WATER TO KEEP FIRE-EXPOSED CONTAINERS COOL. IF LEAK OR SPILL HAS NOT IGNITIED, USE WATER SPRAY TO DISPERSE THE VAPORS AND TO PROVIDE PROTECTION.
Unusual Fire And Expl Hazrds: PRODUCTS OF COMBUSTION MAY CONTAIN CARBON MONOXIDE, CARBON DIOXIDE AND OTHER TOXIC MATERILS. DO NOT ENTER ENCLOSED OR CONFINED SPACE WITHOUT PROPER PPE.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): AVOID HEAT AND FLAME
Materials To Avoid: INCOMATIBLE WITH OXIDIZING MATERIALS.

Health Hazard Data

Precautions for Safe Handling and Use

Control Measures

Transportation Data

Trans Data Review Date: 92325
DOT PSN Code: GJL
DOT Proper Shipping Name: FLAMMABLE LIQUIDS, N.O.S.
DOT Class: 3
DOT ID Number: UN1993
DOT Pack Group: III
DOT Label: FLAMMABLE LIQUID
IMO PSN Code: HIA
IMO Proper Shipping Name: FLAMMABLE LIQUID, N.O.S. o
IMO Regulations Page Number: 3345
IMO UN Number: 1993
IMO UN Class: 3.3
IMO Subsidiary Risk Label: -
IATA PSN Code: MCA
IATA UN ID Number: 1993
IATA Proper Shipping Name: FLAMMABLE LIQUID, N.O.S. *
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: MCA
AFI Prop. Shipping Name: FLAMMABLE LIQUIDS, N.O.S.
AFI Class: 3
AFI ID Number: UN1993
AFI Pack Group: III
AFI Basic Pac Ref: 7-7
MMAC Code: NR
N.O.S. Shipping Name: FLAMMABLE LIQUID, N.O.S
Additional Trans Data: DIESEL FUEL

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 24JUN92
MFR Label Number: NONE
Label Status: G
Common Name: DIESEL FUEL NO. 2
Chronic Hazard: NO
Signal Word: CAUTION!
Acute Health Hazard-Slight: X

Contact Hazard-Slight: X
Fire Hazard-Slight: X
Reactivity Hazard-None: X
Special Hazard Precautions: STORE IN WELL VENTILATED AREEA. KEEP CONTAINER TIGHTLY CLOSED. STORE IN ACCORDANCE WITH NATIONAL FIRE PROTECTION ASSN REGULATIONS. FIRST AID: INHALATION: REMOVE TO FRESH AIR. IF NOT BREATHING, ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN. CALL A CIAN. SKIN: FLUSH SKIN WITH WATER AFTER CONTACT. REMOVE CONTAMINATED THING. EYES: IMMEDIATELY FLUSH WITH WATER FOR 15 MINUTES. CALL A PHYSICIAN. INGESTION: DO NOT INDUCE VOMITING. IMMEDIATELY GIVE TWO GLASSES OF WATER. NEVER GIVE ANYTHING TO IF UNCONCIOUS. CALL MD
Protect Eye: Y
Protect Skin: Y
Label Name: CONOCO INC
Label Street: 600 N DAIRY ASHFORD RD RM 3012
Label P.O. Box: 4784
Label City: HOUSTON
Label State: TX
Label Zip Code: 77210-4784
Label Country: US
Label Emergency Number: 713-293-5550/800-424-9300

SHELL OIL -- 65101 OMALA OIL 68 - PETROLEUM HYDROCARBON: INDUSTRIAL OIL
MATERIAL SAFETY DATA SHEET
NSN: 915000F013983
Manufacturer's CAGE: 54527
Part No. Indicator: B
Part Number/Trade Name: 65101 OMALA OIL 68

General Information

Item Name: PETROLEUM HYDROCARBON: INDUSTRIAL OIL
Company's Name: SHELL OIL CO
Company's Street: 1 SHELL PLZ
Company's P. O. Box: 2463
Company's City: HOUSTON
Company's State: TX
Company's Country: US
Company's Zip Code: 77001-5000
Company's Emerg Ph #: 713-473-9461/713-241-4819
Company's Info Ph #: 713-241-4819/713-473-9461
Record No. For Safety Entry: 002
Tot Safety Entries This Stk#: 002
Status: FE
Date MSDS Prepared: 30MAY91
Safety Data Review Date: 23MAY96
MSDS Preparer's Name: G A VAN GELDER
Preparer's Company: SHELL OIL CO
Preparer's St Or P. O. Box: P O BOX 4320
Preparer's City: HOUSTON
Preparer's State: TX
Preparer's Zip Code: 77210-5000
MSDS Serial Number: BZBXQ

Ingredients/Identity Information

Proprietary: NO
Ingredient: SOLVENT REFINED HYDROTREATED HEAVY PARAFFINIC DISTILLATE, HYDROTREATED HEAVY PARAFFINIC DISTILLATE, LUBRICATING OIL.
Ingredient Sequence Number: 01
Percent: 0-90
NIOSH (RTECS) Number: PY8035500
CAS Number: 64742-54-7
OSHA PEL: 5 MG/CUM (MIST)

Proprietary: NO
Ingredient: CATALYTIC DEWAXED HEAVY PARAFFINIC OIL
Ingredient Sequence Number: 02
Percent: 0-90
NIOSH (RTECS) Number: PY8045500
CAS Number: 64742-70-7

Proprietary: NO
Ingredient: RESIDUAL OILS (PETROLEUM), HYDROTREATED *96-1*
Ingredient Sequence Number: 03
Percent: 5-10
NIOSH (RTECS) Number: 1001346PO
CAS Number: 64742-57-0
ACGIH TLV: 5 MG/CUM

Proprietary: NO
Ingredient: MINOR ADDITIVE
Ingredient Sequence Number: 04
Percent: 2-3
NIOSH (RTECS) Number: 1002892MA

Physical/Chemical Characteristics

Appearance And Odor: YELLOW OIL W/SLIGHT HYDROCARBON ODOR
Melting Point: (SEE SUPP)
Specific Gravity: 0.8899
Solubility In Water: NEGLIGIBLE

Fire and Explosion Hazard Data

Flash Point: 400F
Flash Point Method: COC
Extinguishing Media: WATER FOG, FOAM, DRY CHEMICAL/CO2.
Special Fire Fighting Proc: DON'T ENTER CONFINED FIRE-SPACE W/O FULL BUNKDER GEAR & A POSITIVE PRESSURE NIOSH-APPROVED SCBA. COOL FIRE EXPOSED TAINERS W/WATER.
al Fire And Expl Hazrds: MATERIAL WILL NOT BURN UNLESS PREHEATED. T USE A DIRECT STREAM OF WATER. PRODUCT WILL FLOAT & CAN BE REIGNITED ON SURFACE OF WATER.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): HEAT, OPEN FLAMES
Materials To Avoid: OXIDIZING MATERIALS
Hazardous Decomp Products: THERMAL: CO & OTHER UNIDENTIFIED ORGANIC COMPOUNDS.
Hazardous Poly Occur: NO

Health Hazard Data

Route Of Entry - Inhalation: YES
Route Of Entry - Skin: NO
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: EYES: MINIMAL IRRITATION. SKIN: MILDLY IRRITATING. PROLONGED/REPEATED CONTACT CAN RESULT IN VARIOUS DISORDERS SUCH AS FOLLICULITIS, DERMATITIS & OIL ACNE. INHALATION: VAPORS/OIL MIST MAY CAUSE MILD IRRITATION OF THE RESPIRATORY TRACT. INGESTION: SLIGHTLY TOXIC.
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: NONE
Signs/Symptoms Of Overexp: EYES: MINIMAL IRRITATION. SKIN: MILDLY IRRITATING. PROLONGED/REPEATED CONTACT CAN RESULT IN VARIOUS DISORDERS SUCH AS FOLLICITIES, DERMATITIS & OIL ACNE. INHALATION: VAPORS/OIL MIST MAY CAUSE MILD IRRITATION OF THE RESPIRATORY TRACT. INGESTION: SLIGHTLY TOXIC.
nd Aggravated By Exp: PRE-EXISTING SKIN & RESPIRATORY DISORDERS.
ncy/First Aid Proc: EYES: FLUSH W/WATER FOR 15 MINS. SKIN: WASH W AP & WATER/A WATERLESS HAND CLEANER FOLLOWED BY SOAP & WATER. INHALATION: REMOVE TO FRESH AIR & GIVE OXYGEN IF NEEDED. INGESTION: DON'T IN GENERAL, EMESIS INDUCTION IS UNNECESSARY IN HIGH VISCOSITY, LOW VOLATILITY PRODUCTS, MOST OILS & GREASES.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: LARGE: USE CAUTION WHEN CLEANING. WEAR RESPIRATOR & APPROPRIATE PROTECTIVE CLOTHING. SHUT OFF SOURCE SAFELY. DIKE & CONTAIN. REMOVE W/VACUUM TRUCKS/PUMP TO STORAGE SALVAGE VESSELS. SOAK UP RESIDUE W/AN ABSORBENT. DISPOSE OF PROPERLY. (SUPP)
Waste Disposal Method: DISPOSE OF IAW/FEDERAL, STATE & LOCAL REGULATIONS.
Precautions-Handling/Storing: STORE IN A COOL, DRY PLACE W/ADEQUATE VENTILATION. KEEP AWAY FROM OPEN FLAMES & HIGH TEMPS.
Other Precautions: MINIMIZE SKIN CONTACT.

Control Measures

Respiratory Protection: IF EXPOSURE LIMITS EXCEED, USE A NIOSH-APPROVED RESPIRATOR. USE EITHER AN ATMOSPHERE-SUPPLYING RESPIRATOR/AN AIR-PURIFYING RESPIRATOR FOR ORGANIC VAPORS & PARTICULATES.
Protective Gloves: CHEMICAL RESISTANT/NITRILE
Eye Protection: SAFETY GOGGLES
Other Protective Equipment: OTHER PROTECTIVE CLOTHING AS REQUIRED TO MINIMIZE SKIN CONTACT.

Transportation Data

Disposal Data

Label Data

Label Required: YES
Label Status: G
Common Name: 65101 OMALA OIL 68
Special Hazard Precautions: EYES: MINIMAL IRRITATION. SKIN: MILDLY IRRITATING. PROLONGED/REPEATED CONTACT CAN RESULT IN VARIOUS DISORDERS SUCH AS FOLLICULITIS, DERMATITIS & OIL ACNE. INHALATION: VAPORS/OIL MIST MAY CAUSE MILD IRRITATION OF THE RESPIRATORY TRACT. INGESTION: SLIGHTLY TOXIC. EYES: MINIMAL IRRITATION. SKIN: MILDLY IRRITATING. PROLONGED/REPEATED CONTACT CAN RESULT IN VARIOUS DISORDERS SUCH AS FOLLICITIES, DERMATITIS & OIL ACNE. INHALATION: VAPORS/OIL MIST MAY CAUSE MILD IRRITATION OF THE RESPIRATORY TRACT. INGESTION: SLIGHTLY TOXIC.
Label Name: SHELL OIL CO
Label Street: 1 SHELL PLZ
Label P.O. Box: 2463
Label City: HOUSTON
Label State: TX
Label Zip Code: 77001-5000
Label Country: US
Label Emergency Number: 713-473-9461/713-241-4819

SPECTRUM CHEMICAL MFG -- BENZENE - BENZENE, TECHNICAL
MATERIAL SAFETY DATA SHEET
NSN: 6810002815272
Manufacturer's CAGE: 63415
Part No. Indicator: B
Number/Trade Name: BENZENE

General Information

Item Name: BENZENE, TECHNICAL
Company's Name: SPECTRUM CHEMICAL MFG CORP.
Company's Street: 14422 SOUTH SAN PEDRO STREET
Company's City: GARDENA
Company's State: CA
Company's Country: US
Company's Zip Code: 90248-2027
Company's Emerg Ph #: 800-424-9300(CHEMTREC)/714-864-2310
Company's Info Ph #: 714-864-2310
Distributor/Vendor # 1: CHEMICAL COMMODITIES AGENCY, INC.
Distributor/Vendor # 1 Cage: 60777
Record No. For Safety Entry: 005
Tot Safety Entries This Stk#: 007
Status: SM
Date MSDS Prepared: 01APR97
Safety Data Review Date: 28OCT97
Supply Item Manager: CX
MSDS Serial Number: BYXKC
Hazard Characteristic Code: F5
Unit Of Issue: CN
Unit Of Issue Container Qty: 1 GALLON
Type Of Container: CAN
Net Unit Weight: 7.3 LBS

Ingredients/Identity Information

Proprietary: NO
ent: BENZENE (SARA III)
ent Sequence Number: 01
nt: 100.0
NIOSH (RTECS) Number: CY1400000
CAS Number: 71-43-2
OSHA PEL: 1PPM/5STEL;1910.1028
ACGIH TLV: 10 PPM; A2; 9192
Other Recommended Limit: NONE SPECIFIED

Physical/Chemical Characteristics

Appearance And Odor: COLORLESS TO LIGHT YELLOW WITH AN AROMATIC ODOR
Boiling Point: 176F,80C
Melting Point: 42.0F,5.6C
Vapor Pressure (MM Hg/70 F): 75
Vapor Density (Air=1): 2.8
Specific Gravity: .8765
Evaporation Rate And Ref: 5.1 (BUTYL ACETATE = 1)
Solubility In Water: SLIGHT
Corrosion Rate (IPY): UNKNOWN
Autoignition Temperature: 928F

Fire and Explosion Hazard Data

Flash Point: 12F,-11C
Lower Explosive Limit: 1.2 %
Upper Explosive Limit: 7.8 %
Extinguishing Media: DRY CHEMICAL, CARBON DIOXIDE, WATER SPRAY, REGULAR FOAM
Special Fire Fighting Proc: FIRE FIGHTERS SHOULD WEAR SELF CONTAINED BREATHING APPARATUS AND FULL PROTECTIVE GEAR. COOL TANKS AND CONTAINERS D TO FIRE WITH WATER.
Fire And Expl Hazrds: WITHDRAW IMMEDIATELY IN CASE OF RISING SOUND FROM VENTING DEVISE AND ANY DISCOLORATION OF TANK DUE TO FIRE. AVOID BREATHING HAZARDOUS FUMES/VAPORS.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): HEAT, SPARKS, FLAMES
Materials To Avoid: ACIDS, ARSENIC PENTAFLOURIDE, BROMINE PLUS IRON, CHLORINE, NITRIC ACID, OXYGEN, OZONE, PERCHLORATE
Hazardous Decomp Products: THERMAL DECOMPOSITION PRODUCTS MAY INCLUDE TOXIC OXIDES OF CARBON.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NONE. WILL NOT OCCUR.

Health Hazard Data

LD50-LC50 Mixture: ORAL LD50 (RAT): 930 MG/KG
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: INHALATION CAUSES RESPIRATORY TRACT IRRIATION. SEVERE EXPOSURES MAY RESULT IN PULMONARY EDEMA, NAUSEA, VOMITING, HEADACHE, DIZZINESS, DROWSINESS, EUPHORIA, IRRITABILITY, WEAK/ RAPID PULSE, CYANOSIS OF LIPS & TINNITUS, CARDIAC ARRYTHMIA, RENAL CONGESTION, DEATH. KNOWN HUMAN CARCINOGEN.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: CONTAINS Benzene [71-43-2] WHICH IS LISTED BY NTP AND IARC AND REGULATED BY OSHA AS A CARCINOGEN.
Signs/Symptoms Of Overexp: MODERATELY TOXIC BY INHALATION AND INGESTION. CENTRAL NERVOUS SYSTEM DEPRESSANT, POISONING MAY ALSO EFFECT THE IMMUNE SYSTEM AND THE HEART. USE OF ALCOHOLIC BEVERATES MAY ENHANCE THE TOXIC EFFECTS. USE OF STIMULANTS SUCH AS EPINEPHRINE MAY CAUSE CARDIAC ARRHYMIAS. SKIN BLISTERING, DERMATITIS, EYE IRRITATION.
Med Cond Aggravated By Exp: PERSONS WITH CERTAIN IMMUNOLOGICAL TENDENCIES, POOR NUTRITION, ANEMIA, AND DRUG OR CHEMICALLY INDUCED AGRANULOCYTEMIA.
Emergency/First Aid Proc: INHALATION: REMOVE TO FRESH AIR. IF BREATHING HAS STOPPED GIVE ARTIFICIAL RESPIRATORION. GIVE OXYGEN IF AVAILABLE. GET MEDICAL ATTENTION. SKIN: WASH AREA WITH SOAP AND WATER. WASH CONTAMINATED CLOTHING. GET IMMEDIATE MEDICAL ATTENTION. EYES: FLOOD WITH WATER OR NORMAL SALINE FOR 15 MINUTES. GET MEDICAL ATTENTION. INGESTION: USE EXTREME CARE TO PREVENT ASPIRATION. GET IMMEDIATE MEDICAL ATTENTION.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: SHUT OFF IGNITION SOURCES. USE WATER SPRAY TO REDUCE VAPORS. TAKE UP WITH SAND OR OTHER ABSORBENT MATERIAL. PLACE INTO CONTAINERS FOR LATER DISPOSAL. APPLY ACTIVATED CARBON AT TEN TIMES THE SPILL AMOUNT IN REGION OF 10 PPM.
Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.
Waste Disposal Method: DISPOSAL IN ACCORDANCE WITH STANDARDS TO GENERATORS OF HAZARDOUS WASTE,4 CFR 262 EPA HAZARDOUS WASTE NUMBER D019, OBSERVE ALL FEDERAL,STATE AND LOCAL REGULATIONS WHEN DISPOSING OF THIS SUBSTANCE.
Precautions-Handling/Storing: BONDING AND GROUNDING GUIDELINES NEED TO BE MET AS SPECIFIED IN NFPA 77-1983 ON STATIC ELECTRICITY. DO NOT BREATHE GAS, FUMES, VAPORS.
Other Precautions: AVOID CONTACT WITH HEAT, SPARKS, FLAMES OR OTHER SOURCES OF IGNITION.

Control Measures

Respiratory Protection: BASED UPON CONTMINATION LEVELS IN THE WORK PLACE. FOR EXAMPLE: AIR PURIFYING RESIPRATOR WITH ORGANIC VAPOR CARTRIDGE. GREATER CONCENTRATIONS: SELF-CONTAINED BREATHING APPARATUS.
Ventilation: PROVIDE EXHAUST OR PROCESS ENCLOSURE VENTICATION TO MEET PUBLISHED EXPOSURE LIMITS EQUIPMENT MUST BE EXPLOSION PROOF.
Protective Gloves: IMPERVIOUS
Eye Protection: SAFETY GOGGLES UNLESS RESPIRATOR WORN.
Other Protective Equipment: CHEMICAL RESISTANT CLOTHING AS NECESSARY TO PREVENT SKIN CONTACT. AN EMERGENCY EYEWASH AND SHOWER SHOULD BE AVAILABLE.
Work Hygienic Practices: WASH THOROUGHLY AFTER HANDLING AND BEFORE EATING, SMOKING OR USING SANTIARY FACILITIES.
Suppl. Safety & Health Data: NONE SPECIFIED BY MANUFACTURER.

Transportation Data

Trans Data Review Date: 96129
DOT PSN Code: BRS
DOT Proper Shipping Name: BENZENE
DOT Class: 3
DOT ID Number: UN1114
Pack Group: II
Label: FLAMMABLE LIQUID
PSN Code: BXB
IMO Proper Shipping Name: BENZENE
IMO Regulations Page Number: 3185
IMO UN Number: 1114
IMO UN Class: 3.2
IMO Subsidiary Risk Label: -
IATA PSN Code: DBA
IATA UN ID Number: 1114
IATA Proper Shipping Name: BENZENE
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: DBA
AFI Symbols: 0
AFI Prop. Shipping Name: BENZENE
AFI Class: 3
AFI ID Number: UN1114
AFI Pack Group: II
AFI Basic Pac Ref: A7.3

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 08MAY96
MFR Label Number: NONE
Label Status: F
Common Name: BENZENE
ic Hazard: YES
. Word: DANGER!
te Health Hazard-Severe: X
Contact Hazard-Slight: X
Fire Hazard-Severe: X
Reactivity Hazard-None: X
Special Hazard Precautions: CENTRAL NERVOUS SYSTEM DEPRESSANT; BONE MARROW DEPRESSANT. POISONING MAY EFFECT THE IMMUNE SYSTEM. INHALATION- RESPIRATORY TRACT IRRITATION. PULMONARY EDEMA. DEATH DUE TO ASPHXIA EYES- IRRITATION. FIRST AID: INHALATION- REMOVE TO FRESH AIR GIVE ARTIFICIAL RESPIRATION IF NEEDED. SEEK MEDICAL ATTENTION. SKIN- REMOVE CONTAMINATED SHOES AND CLOTHING. WASH AFFECTED AREA WITH SOAP AND WATER. GET MEDICAL HELP. EYES- FLUSH WITH COPIOUS AMOUNTS OF NORMAL SALINE OR WATER WITHIN 15 MINUTES. GET IMMEDIATE MEDICAL ATTENTION.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: SPECTRUM CHEMICAL MANUFACTURING CORP.
Label Street: 14422 SOUTH SAN PEDRO STREET
Label City: GARDENA
Label State: CA
Label Zip Code: 90248-2027
Label Country: US
Label Emergency Number: 714-864-2310/213-516-8000

SCIENCE KIT -- TOLUENE - TOLUENE
MATERIAL SAFETY DATA SHEET
NSN: 681000D005045
Manufacturer's CAGE: 57020
Part No. Indicator: A
Part Number/Trade Name: TOLUENE

General Information

Item Name: TOLUENE
Company's Name: SCIENCE KIT INC
Company's Street: 777 E PARK DR
Company's City: TONAWANDA
Company's State: NY
Company's Country: US
Company's Zip Code: 14150-6708
Company's Emerg Ph #: 800-424-9300(CHEMTREC)
Company's Info Ph #: 800-256-2586
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 01DEC94
Safety Data Review Date: 10JUN96
Supply Item Manager: CX
MSDS Preparer's Name: R.M.CARLSON
MSDS Serial Number: BZPCL
Specification Number: NONE
Spec Type, Grade, Class: NONE
Hazard Characteristic Code: F3
Unit Of Issue: NK
Unit Of Issue Container Qty: UNKNOWN
Type Of Container: UNKNOWN
Net Unit Weight: UNKNOWN

Ingredients/Identity Information

Proprietary: NO
Ingredient: TOLUENE (SARA 313) (CERCLA)
Ingredient Sequence Number: 01
Percent: MAJOR
NIOSH (RTECS) Number: XS5250000
CAS Number: 108-88-3
OSHA PEL: 200 PPM; Z-2
ACGIH TLV: S, 50 PPM; 9596
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: CLEAR COLORLESS LIQUID,AROMATIC BENZENE-LIKE ODOR.
Boiling Point: 232F,111C
Melting Point: -139F,-95C
Vapor Pressure (MM Hg/70 F): 22
Vapor Density (Air=1): 3.14
Specific Gravity: 0.86
Decomposition Temperature: UNKNOWN
Evaporation Rate And Ref: 4.5 ETHER=1
Solubility In Water: VIRTUALLY INSOLUBLE
Percent Volatiles By Volume: 100
Corrosion Rate (IPY): UNKNOWN
Autoignition Temperature: 896F

Fire and Explosion Hazard Data

Flash Point: 39.2F,4.0C
Flash Point Method: CC
Lower Explosive Limit: 1.2
Upper Explosive Limit: 7.1
Extinguishing Media: DRY CHEMICAL,CARBON DIOXIDE,FOAM.
Special Fire Fighting Proc: USE A SELF-CONTAINED BREATHING APPARATUS AND FULL PROTECTIVE EQUIPMENT.COOL FIRE EXPOSED CONTAINERS WITH WATER FOG.
Unusual Fire And Expl Hazrds: VAPORS ARE HEAVIER THAN AIR,CAN TRAVEL DISTANCES ALONG THE GROUND,AND FLASHBACK AT THE SOURCE.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): HIGH HEAT,SOURCES OF IGNITION.
Materials To Avoid: STRONG OXIDIZERS,NITRIC AND SULFURIC ACIDS,STRONG
'S,SOME PLASTICS,RUBBERS,COATING AND SOME AMINES.
lous Decomp Products: CARBON DIOXIDE,CARBON MONOXIDE.
ardous Poly Occur: NO

Health Hazard Data

LD50-LC50 Mixture: TLV IS 100PPM (TWA)
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: EYES:MAY CAUSE IRRITATION.SKIN:MAY CAUSE IRRITATION.INGEST:MAY CAUSE GI TRACT IRRITATION.INHAL:MAY CAUSE RESPIRATORY IRRITATION AND CNS DEPRESSION.CHRONIC:DERMATITIS,LIVER,KIDNEY AND BLOOD DISORDERS.
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: THERE ARE NO INGREDIENTS ABOVE 0.1% WHICH ARE IDENTIFIED AS CARCINOGENS BY NTP,IARC OR OSHA.
Signs/Symptoms Of Overexp: NAUSEA,HEADACHE,DIZZINESS,DROWSINESS.
Med Cond Aggravated By Exp: PERSONS WITH PRE-EXISTING SKIN,EYE,KIDNEY, LIVER AND RESPIRATORY AILMENTS MAY BE AT INCREASED RISK FROM EXPOSURE.
Emergency/First Aid Proc: SKIN:REMOVE CONTAMINATED CLOTHING;WASH WITH SOAP OR MILD DETERGENT AND WATER.GET MEDICAL ATTENTION IF IRRITATION PERSISTS.EYES:FLUSH WITH WATER FOR 15 MINUTES.GET IMMEDIATE MEDICAL ATTENTION.INHAL:REMOVE TO FRESH AIR.GIVE OXYGEN OR ARTIFICIAL RESPIRATION IF NEEDED.INGEST:DO NOT INDUCE VOMITING.GET PROMPT QUALIFIED MEDICAL ATTENTION.IF CONSCIOUS,GIVE WATER OR MILK TO DRINK.

Precautions for Safe Handling and Use

f Matl Released/Spill: ELIMINATE SOURCES OF IGNITION.USE PROPER
.RATORY AND PROTECTIVE EQUIPMENT.SHUT OFF LEAK IF SAFE.DIKE.SOAK UP
H A NON-COMBUSTIBLE INERT ABSORBANT(CLAY,SAND);PLACE IN PROPER CONTAINER FOR DISPOSAL.WASH SPILL AREA TO REMOVE SLIPPINESS.
Neutralizing Agent: NOT APPLICABLE.
Waste Disposal Method: DISPOSE OF IN ACCORDANCE WITH FEDERAL,STATE AND LOCAL REGULATIONS.DO NOT FLUSH TO SEWER.
Precautions-Handling/Storing: STORE IN A COOL,DRY,WELL-VENTILATED PLACE. KEEP CONTAINER CLOSED WHEN NOT IN USE.KEEP AWAY FROM HEAT,SPARKS,FLAMES AND INCOMPATIBLE MATERIALS.
Other Precautions: FOLLOW LABEL DIRECTIONS.AVOID BREATHING VAPORS.AVOID SKIN AND EYE CONTACT.

Control Measures

Respiratory Protection: WHERE ENVIRONMENTAL CONTROLS ARE LACKING OR IN ENCLOSED SPACES USE EITHER A SELF-CONTAINED BREATHING APPARATUS OR A NIOSH/ MSHA APPROVED RESPIRATOR FOR ORGANIC VAPORS,DEPENDING ON THE AIRBORN CONCENTRATION.
Ventilation: LOCAL VENTILATION AT THE WORKSITE;MECHANICAL(GENERAL) VENTILATION TO MAINTAIN TLV/PEL.USE NON-SPARKING EQUIPMENT.
Protective Gloves: VINYL OR RUBBER.
Eye Protection: CHEMICAL SPLASH GOGGLES
Other Protective Equipment: LAB COAT.PROVIDE A LOCAL EYE WASH STATION AND SAFETY SHOWER.
Work Hygienic Practices: EXECISE GOOD LABORATORY PRACTICES.WASH HANDS AFTER USE AND BEFORE EATING.
Suppl. Safety & Health Data: NONE

Transportation Data

ata Review Date: 96162
SN Code: OJY
DOT Proper Shipping Name: TOLUENE
DOT Class: 3
DOT ID Number: UN1294

DOT Pack Group: II
DOT Label: FLAMMABLE LIQUID
IMO PSN Code: OSR
IMO Proper Shipping Name: TOLUENE
IMO Regulations Page Number: 3285
IMO UN Number: 1294
IMO UN Class: 3.2
IMO Subsidiary Risk Label: -
IATA PSN Code: YEL
IATA UN ID Number: 1294
IATA Proper Shipping Name: TOLUENE
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: YEL
AFI Prop. Shipping Name: TOLUENE
AFI Class: 3
AFI ID Number: UN1294
AFI Pack Group: II
AFI Basic Pac Ref: A7.3
MMAC Code: NR
Additional Trans Data: NONE

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 10JUN96
MFR Label Number: UNKNOWN
Label Status: F
Common Name: TOLUENE
Signal Word: WARNING!
Acute Health Hazard-Slight: X
Contact Hazard-Slight: X
Fire Hazard-Moderate: X
Reactivity Hazard-None: X
Special Hazard Precautions: EYES:MAY CAUSE IRRITATION.SKIN:MAY CAUSE IRRITATION.INGEST:MAY CAUSE GI TRACT IRRITATION.INHAL:MAY CAUSE RESPIRATORY IRRITATION AND CNS DEPRESSION.CHRONIC:DERMATITIS,LIVER,KIDNEY AND BLOOD DISORDERS. FIRST AID: SKIN:REMOVE CONTAMINATED CLOTHING;WASH WITH SOAP OR DO NOT INDUCE VOMITING.GET PROMPT QUALIFIED MEDICAL ATTENTION.IF CONSCIOUS, GIVE WATER OR MILK TO DRINK.
Protect Eye: Y
Protect Skin: Y
Label Name: SCIENCE KIT INC
Label Street: 777 E PARK DR
Label City: TONAWANDA
Label State: NY
Label Zip Code: 14150-6708
Label Country: US
Label Emergency Number: 800-424-9300(CHEMTREC)
Year Procured: 1995

CHEM SERVICE – 0-770, ETHYLBENZENE
MATERIAL SAFETY DATA SHEET
NSN: 681000N033034
Manufacturer's CAGE: 8Y898
Part No. Indicator: A
Number/Trade Name: 0-770, ETHYLBENZENE

General Information

Company's Name: CHEM SERVICE INC
Company's Street: 660 TOWER LANE
Company's P. O. Box: 3108
Company's City: WEST CHESTER
Company's State: PA
Company's Country: US
Company's Zip Code: 19381-3108
Company's Emerg Ph #: 215-692-3026
Company's Info Ph #: 215-692-3026
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 002
Status: SMJ
Date MSDS Prepared: 16MAR92
Safety Data Review Date: 28AUG95
MSDS Serial Number: BPLSP
Hazard Characteristic Code: F3

Ingredients/Identity Information

Proprietary: NO
Ingredient: BENZENE, ETHYL-;(ETHYLBENZENE)
Ingredient Sequence Number: 01
NIOSH (RTECS) Number: DA0700000
CAS Number: 100-41-4
OSHA PEL: 100 PPM, 125 STEL
ACGIH TLV: 100 PPM, 125 STEL

Physical/Chemical Characteristics

Appearance And Odor: COLORLESS LIQUID WITH AN AROMATIC ODOR
Boiling Point: 277F,136C
Melting Point: -139F,-95C
Vapor Pressure (MM Hg/70 F): 7.1 @20C
Vapor Density (Air=1): 0.887
Solubility In Water: INSOLUBLE

Fire and Explosion Hazard Data

Flash Point: 71.6F,22C
Lower Explosive Limit: 1%
Upper Explosive Limit: 6.7%
Extinguishing Media: CARBON DIOXIDE, DRY CHEMICAL POWDER OR SPRAY.
Special Fire Fighting Proc: WEAR NIOSH/MSHA APPROVED SCBA & FULL PROTECTIVE EQUIPMENT (FP N).
Unusual Fire And Expl Hazrds: NONE SPECIFIED BY MANUFACTURER.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MANUFACTURER.
Materials To Avoid: STRONG OXIDIZING AGENTS.
Hazardous Decomp Products: EMITS TOXIC FUMES UNDER FIRE CONDITIONS.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT RELEVANT.

Health Hazard Data

LD50-LC50 Mixture: LD50:(ORAL RAT):3500 MG/KG.
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: CAN CAUSE SKIN AND EYE IRRITATION. MAY BE HARMFUL IF ABSORBED THROUGH SKIN. MAY BE HARMFUL IF INHALED. MAY BE HARMFUL

IF SWALLOWED. CAN BE IRRITATING TO MUCOUS MEMBRANES. PROLONGED EXPOSURE MAY CAUSE NAUSEA, HEADACHE, DIZZINESS AND/OR EYE DAMAGE. CAN CAUSE NERVOUS SYSTEM INJURY. DUST &/OR VAPORS (EFTS OF OVEREXP)
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: NOT RELEVANT.
Signs/Symptoms Of Overexp: HLTH HAZ:CAN CAUSE IRRITATION TO RESPIRATORY TRACT.
Med Cond Aggravated By Exp: NONE SPECIFIED BY MANUFACTURER.
Emergency/First Aid Proc: EYE:FLUSH CONTINUOUSLY WITH WATER FOR 15-20 MINUTES. SKIN:FLUSH WITH WATER FOR 15-20 MINUTES. IF NO BURNS HAVE OCCURED-USE SOAP & WATER TO CLEANSE SKIN. INHAL:MOVE TO FRESH AIR. GIVE OXYGEN IF PATIENT IS HAVING DIFFICULTY BREATHING. IF PATIENT STOPPED BREATHING, GIVE ARTF RESP. IF PATIENT IS IN CARDIAC ARREST GIVE CPR. CONTINUE LIFE SUPPORTING MEASURES UNTIL MD ARRIVES. INGEST:CALL MD IMMED(FP N)

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: EVACUATE AREA. WEAR APPROPRIATE OSHA REGULATED EQUIPMENT. VENTILATE AREA. ABSORB ON VERMICULITE OR SIMILAR MATERIAL. SWEEP UP AND PLACE IN AN APPROPRIATE CONTAINER. HOLD FOR DISPOSAL. WASH CONTAMINATED SURFACES TO REMOVE ANY RESIDUES.
Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.
Waste Disposal Method: BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER. DISPOSAL MUST BE IN ACCORDANCE WITH LOCAL, STATE AND FEDERAL REGULATIONS (FP N).
Precautions-Handling/Storing: KEEP TIGHTLY CLOSED IN A COOL, DRY PLACE. STORE ONLY WITH COMPATIBLE MATERIALS.
Other Precautions: AVOID CONTACT WITH SKIN, EYES AND CLOTHNG. ALL CHEMICALS SHOULD BE CONSIDERED HAZARDOUS-AVOID DIRECT PHYSICAL CONTACT.

Control Measures

Respiratory Protection: NIOSH/MSHA APPROVED RESPIRATOR APPROPRIATE FOR EXPOSRRE OF CONCERN (FP N).
Ventilation: THIS CHEMICAL SHOULD ONLY BE HANDLED IN A HOOD.
Protective Gloves: IMPERVIOUS GLOVES (FP N).
Eye Protection: CHEMICAL WORKERS GOGGLES (FP N).
Other Protective Equipment: NONE SPECIFIED BY MANUFACTURER.
Work Hygienic Practices: CONTACT LENSES SHOULD NOT BE WORN.
Suppl. Safety & Health Data: NONE SPECIFIED BY MANUFACTURER.

Transportation Data

Trans Data Review Date: 92363
DOT PSN Code: FYP
DOT Proper Shipping Name: ETHYLBENZENE
DOT Class: 3
DOT ID Number: UN1175
DOT Pack Group: II
DOT Label: FLAMMABLE LIQUID
IMO PSN Code: GQL
IMO Proper Shipping Name: ETHYLBENZENE
IMO Regulations Page Number: 3222
IMO UN Number: 1175
IMO UN Class: 3.2
IMO Subsidiary Risk Label: -
IATA PSN Code: LCB
IATA UN ID Number: 1175
IATA Proper Shipping Name: ETHYLBENZENE
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: LCB
AFI Prop. Shipping Name: ETHYLBENZENE
AFI Class: 3
AFI ID Number: UN1175
AFI Pack Group: II
AFI Basic Pac Ref: 7-7

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 22JUL92
Label Date: 22JUL92
Label Status: G
on Name: 0-770, ETHYLBENZENE
c Hazard: NO
nal Word: DANGER!
Acute Health Hazard-Moderate: X
Contact Hazard-Moderate: X
Fire Hazard-Severe: X
Reactivity Hazard-None: X
Special Hazard Precautions: FLAMMABLE-POISON! STORE IN A COOL, DRY PLACE. ACUTE:CAN CAUSE SKIN/EYE IRRITATION. MAY BE HARMFUL IF ABSORBED THROUGH SKIN. MAY BE HARMFUL IF INHALED/SWALLOWED. CAN BE IRRITATING TO MUCOUS MEMBRANES. PROLONGED EXPOSURE MAY CAUSE NAUSEA, HEADACHE, DIZZINESS AND/OR EYE DAMAGE. CAN CAUSE NERVOUS SYSTEM INJURY. DUST AND/OR VAPORS CAN CAUSE IRRITATION TO RESPIRATORY TRACT. CHRONIC:NONE LISTED BY MANUFACTURER.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: CHEM SERVICE INC.
Label Street: 660 TOWER LANE
Label P.O. Box: 3108
Label City: WEST CHESTER
Label State: PA
Label Zip Code: 19381-3108
Label Country: US
Label Emergency Number: 215-692-3026

MOBIL OIL -- XYLENE - XYLENE,TECHNICAL
MATERIAL SAFETY DATA SHEET
NSN: 6810005844070
Manufacturer's CAGE: 3U728
Part No. Indicator: B
Part Number/Trade Name: XYLENE

General Information

Item Name: XYLENE,TECHNICAL
Company's Name: MOBIL OIL CORP
Company's Street: 3225 GALLOWS RD
Company's City: FAIRFAX
Company's State: VA
Company's Country: US
Company's Zip Code: 22037
Company's Emerg Ph #: 609-737-4411 800-424-9300(CHEMTREC)
Distributor/Vendor # 1: MAGNOLIA CHEMICAL (504-733-6600)
Distributor/Vendor # 1 Cage: 0LNB7
Record No. For Safety Entry: 006
Tot Safety Entries This Stk#: 017
Status: FE
Date MSDS Prepared: 05MAR96
Safety Data Review Date: 19MAR98
Supply Item Manager: CX
MSDS Preparer's Name: UNKNOWN
MSDS Serial Number: CGLDB
Specification Number: TT-X-916 (FED SPEC)
Spec Type, Grade, Class: ASTM D846
Hazard Characteristic Code: F4
Unit Of Issue: CN
Unit Of Issue Container Qty: 5 GALLONS
Type Of Container: CAN
Net Unit Weight: 36.0 LBS

Ingredients/Identity Information

Proprietary: NO
Ingredient: XYLENES (O-,M-,P- ISOMERS) (SARA 313) (CERCLA)
Ingredient Sequence Number: 01
Percent: 100
NIOSH (RTECS) Number: ZE2100000
CAS Number: 1330-20-7
OSHA PEL: 100 PPM
ACGIH TLV: 100 PPM/150STEL;9596
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: LIQUID;COLORLESS;XYLENE ODOR.
Boiling Point: 309F,154C
Vapor Pressure (MM Hg/70 F): 5.1
Vapor Density (Air=1): 3.6
Specific Gravity: 0.863
Decomposition Temperature: UNKNOWN
Solubility In Water: NEGLIGIBLE
Percent Volatiles By Volume: 100
Viscosity: <1CST@40C
Corrosion Rate (IPY): UNKNOWN

Fire and Explosion Hazard Data

Flash Point: 80.6F,27.0C
Lower Explosive Limit: 1.1
Upper Explosive Limit: 7
Extinguishing Media: CARBON DIOXIDE,FOAM,DRY CHEMICAL,WATER FOG.
Special Fire Fighting Proc: USE A SELF-CONTAINED BREATHING APPARATUS. COOL FIRE EXPOSED CONTAINERS WITH WATER SPRAY.AVOID POLLUTION OF WATERWAYS FROM RUNOFF.
Unusual Fire And Expl Hazrds: FLAMMABLE VAPORS.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): STATIC BUILDUP,HIGH HEAT AND SOURCES OF IGNITION.
Materials To Avoid: STRONG OXIDIZING AGENTS,ACTIVE METALS,HALOGENS,ACIDS.
Hazardous Decomp Products: CARBON DIOXIDE,CARBON MONOXIDE
·dous Poly Occur: NO

Health Hazard Data

LD50-LC50 Mixture: ORAL LD50 (RAT) IS 4300MG/KG
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: INGEST:PRACTICALLY NON-TOXIC;>2G/KG. ASPIRATION HAZARD.TINHAL:HARMFUL IF INHALED.EYES:IRRITANT.SKIN-PRACTICALLY NON-IRRITATING,BUT MAY CAUSE DEFATTING.
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: PARA-XYLENE IS THE MOST FETOTOXIC ISOMER.
Signs/Symptoms Of Overexp: POSSIBLE DERMATITIS.
Med Cond Aggravated By Exp: NONE SPECIFIED BY MFR.
Emergency/First Aid Proc: EYES:FLUSH WITH WATER.GET MEDICAL ASSISTANCE.SKIN:WASH WITH SOAP AND WATER.INHAL:REMOVE FROM EXPOSURE.CALL PHYSICIAN,GIVE OXYGEN OR MOUTH-TO-MOUTH RESCUSITATION IF NEEDED.INGEST:DO NOT INDUCE VOMITING.GET PROMPT QUALIFIED MEDICAL ASSISTANCE.IF CONSCIOUS, GIVE 1-2 GLASSES OF WATER.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: ELIMINATE SOURCES OF IGNITION.VENTILATE AREA.SOAK UP WITH A NON-COMBUSTIBLE ABSORBANT.PLACE IN AN APPROPRIATE CONTAINER FOR DISPOSAL.PREVENT POLLUTION OF WATERWAYS;CALL 800-424-8802 FOR SPILL REPORTING.
Neutralizing Agent: NOT APPLICABLE
W··ste Disposal Method: DISPOSE OF IN ACCORDANCE WITH FEDERAL,STATE AND REGULATIONS.THIS ITEM MAY BE CLASSIFIED AS RCRA HAZARDOUS V .(IGNITABILITY,CORROSIVITY,REACTIVITY,TCLP).
P utions-Handling/Storing: STORE IN A COOL,DRY PLACE.AVOID HEAT,SPARKS, FLAMES AND INCOMPATIBLE MATERIALS.KEEP CONTAINERS CLOSED.
Other Precautions: GROUND OR BOND CONTAINERS WHEN TRANSFERRING LIQUIDS.

Control Measures

Respiratory Protection: USE A NIOSH/MSHA APPROVED RESPIRATOR FOR ORGANIC VAPOR.
Ventilation: USE LOCAL EXHAUST AT THE WORKSITE;DILUTION VENTILATION TO MAINTAIN THE TLV/PEL.
Protective Gloves: IMPERVIOUS.
Eye Protection: CHEMICAL SPLASH GOGGLES.
Other Protective Equipment: PROTECTIVE CLOTHING AS NEEDED.PROVIDE AN EYE WASH STATION AND QUICK DRENCH SHOWER.
Work Hygienic Practices: USE REASONABLE CARE IN HANDLING THIS PRODUCT.WASH HANDS AFTER HANDLING MATL AND BEFORE EATING.
Suppl. Safety & Health Data: NONE

Transportation Data

Trans Data Review Date: 98078
DOT PSN Code: PWS
DOT Proper Shipping Name: XYLENES
DOT Class: 3
DOT ID Number: UN1307
DOT Pack Group: III
DOT Label: FLAMMABLE LIQUID
IMO PSN Code: PPF
I··· Proper Shipping Name: XYLENES
I ulations Page Number: 3394
IM Number: 1307
IMO UN Class: 3.3
IMO Subsidiary Risk Label: -
IATA PSN Code: ZPL

IATA UN ID Number: 1307
IATA Proper Shipping Name: XYLENES
IATA UN Class: 3
IATA Label: FLAMMABLE LIQUID
AFI PSN Code: ZPL
AFI Prop. Shipping Name: XYLENES
AFI Class: 3
AFI ID Number: UN1307
AFI Pack Group: III
AFI Special Prov: P5
AFI Basic Pac Ref: A7.3
MMAC Code: NR
Additional Trans Data: RQ FOR XYLENE IS 100 LBS.

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 19MAR98
MFR Label Number: UNKNOWN
Label Status: F
Common Name: XYLENE
Chronic Hazard: NO
Signal Word: WARNING!
Acute Health Hazard-Slight: X
Contact Hazard-Slight: X
Fire Hazard-Moderate: X
Reactivity Hazard-None: X
Special Hazard Precautions: INGEST:PRACTICALLY NON-TOXIC;>2G/KG.ASPIRATION HAZARD.TINHAL:HARMFUL IF INHALED.EYES:IRRITANT.SKIN-PRACTICALLY NON-IRRITATING,BUT MAY CAUSE DEFATTING. STORE IN A COOL,DRY PLACE.AVOID HEAT, EYES:FLUSH WITH WATER.GET MEDICAL ASSISTANCE.SKIN:WASH WITH SOAP AND WATER. INHAL:REMOVE FROM EXPOSURE.CALL PHYSICIAN,GIVE OXYGEN OR MOUTH-TO-MOUTH RESCUSITATION IF NEEDED.INGEST:DO NOT INDUCE VOMITING.GET PROMPT QUALIFIED MEDICAL ASSISTANCE.IF CONSCIOUS,GIVE 1-2 GLASSES OF WATER.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: MOBIL OIL CORP
Label Street: 3225 GALLOWS RD
Label City: FAIRFAX
Label State: VA
Label Zip Code: 22037
Label Country: US
Label Emergency Number: 609-737-4411 800-424-9300(CHEMTREC)

ULTRA SCIENTIFIC -- US-116 POLYNUCLEAR AROMATIC HYDROCARBONS MIXTURE
MATERIAL SAFETY DATA SHEET
NSN: 681000F037641
Manufacturer's CAGE: 0MU35
Part No. Indicator: A
Part Number/Trade Name: US-116 POLYNUCLEAR AROMATIC HYDROCARBONS MIXTURE

General Information

Item Name: AT 2000 UG/ML IN METHYLENE CHLORIDE/BENZENE
Company's Name: ULTRA SCIENTIFIC
Company's Street: 250 SMITH STREET
Company's City: NORTH KINGSTOWN
Company's State: RI
Company's Country: US
Company's Zip Code: 02852-5000
Company's Emerg Ph #: 401-294-9400
Company's Info Ph #: 401-294-9400
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 15AUG94
Safety Data Review Date: 13DEC94
Preparer's Company: ULTRA SCIENTIFIC
Preparer's St Or P. O. Box: 250 SMITH STREET
Preparer's City: NORTH KINGSTOWN
Preparer's State: RI
Preparer's Zip Code: 02852-5000
MSDS Serial Number: BWJNN

Ingredients/Identity Information

Proprietary: NO
Ingredient: DICHLOROMETHANE (METHYLENE CHLORIDE) (SUSP HUMAN CARC BY ACGIH, SUSP ANIM CARC BY IARC; NTP - IARC GROUP 2B) *94-4*
Ingredient Sequence Number: 01
Percent: 49.82
NIOSH (RTECS) Number: PA8050000
CAS Number: 75-09-2
ACGIH TLV: 174 MG/CUM (A2)

Proprietary: NO
Ingredient: BENZENE (SUSPECTED HUMAN CARC BY ACGIH, IARC, SUSPECTED ANIMAL CARC BY IARC, CARCINOGEN BY NTP - GROUP 1) *94-4*
Ingredient Sequence Number: 02
Percent: 49.82
NIOSH (RTECS) Number: CY1400000
CAS Number: 71-43-2
ACGIH TLV: 0.3 MG/CUM (A2) IC
Other Recommended Limit: 16 MG/CUM

Proprietary: NO
Ingredient: 7,12-DIMETHYLBENZ-A!ANTHRACENE
Ingredient Sequence Number: 03
Percent: 0.182
NIOSH (RTECS) Number: CW3850000
CAS Number: 57-97-6

Proprietary: NO
Ingredient: 3-METHYLCHOLANTHRENE
Ingredient Sequence Number: 04
Percent: 0.182
NIOSH (RTECS) Number: FZ3675000
CAS Number: 56-49-5

Physical/Chemical Characteristics

Appearance And Odor: LIQUID

Fire and Explosion Hazard Data

Extinguishing Media: CO2, DRY CHEMICAL POWDER, WATER SPRAY

Reactivity Data

Stability: YES
Materials To Avoid: STRONG OXIDIZERS
Hazardous Poly Occur: NO

Health Hazard Data

LD50-LC50 Mixture: ORAL LD50 (RAT): 2136 MG/KG (SEE SUPP)
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: NO
Route Of Entry - Ingestion: NO
Health Haz Acute And Chronic: TOXIC, IRRITATION.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: SEE INGREDIENTS
Signs/Symptoms Of Overexp: IRRITATION.
Emergency/First Aid Proc: EYES/SKIN: FLUSH W/COPIOUS AMOUNTS OF WATER. INHALATION: REMOVE TO FRESH AIR. GIVE OXYGEN, IF NEEDED. OBTAIN MEDICAL ATTENTION IN ALL CASES.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: A LEAKING BOTTLE MAY BE PLACED IN A PLASTIC BAG & NORMAL DISPOSAL PROCEDURES FOLLOWED. LIQUID SAMPLES MAY BE ABSORBED ON VERMICULITE/SAND.
Waste Disposal Method: BURN IN A CHEMICAL INCINERATOR EQUIPPED W/AN AFTERBURNER & SCRUBBER. DISPOSE OF IAW/FEDERAL, STATE & LOCAL REGULATIONS.
Precautions-Handling/Storing: USE APPROPRIATE OSHA/MSHA APPROVED SAFETY EQUIPMENT. KEEP TIGHTLY CLOSED & STORE IN A COOL, DRY PLACE.
Other Precautions: THIS MATERIAL SHOULD ONLY BE USED BY THOSE PERSONS TRAINED IN THE SAFE HANDLING OF HAZARDOUS CHEMICALS.

Control Measures

Protective Gloves: REQUIRED
Eye Protection: CHEMICAL GOGGLES, FACESHIELD
Other Protective Equipment: CHEMICAL RESISTANT CLOTHING, LAB COAT/RUBBER APRON.
Suppl. Safety & Health Data: ORAL LD50 INFORMATION IS FOR METHYLENE CHLORIDE.

Transportation Data

Disposal Data

Label Data

Label Required: YES
Label Status: G
Common Name: US-116 POLYNUCLEAR AROMATIC HYDROCARBONS MIXTURE
Special Hazard Precautions: TOXIC, IRRITATION. IRRITATION.
Label Name: ULTRA SCIENTIFIC
Label Street: 250 SMITH STREET
Label City: NORTH KINGSTOWN
Label State: RI
Label Zip Code: 02852-5000
Label Country: US
Label Emergency Number: 401-294-9400

GFS CHEMICALS -- CADMIUM, 1147
MATERIAL SAFETY DATA SHEET
NSN: 535000N058818
Manufacturer's CAGE: 0TNM0
Part No. Indicator: A
Number/Trade Name: CADMIUM, 1147

General Information

Company's Name: GFS CHEMICALS INC
Company's P. O. Box: 245
Company's City: POWELL
Company's State: OH
Company's Country: US
Company's Zip Code: 43065
Company's Emerg Ph #: 800-858-9682
Company's Info Ph #: 800-858-9682
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SMJ
Date MSDS Prepared: 24AUG92
Safety Data Review Date: 05MAY95
MSDS Preparer's Name: L M
Preparer's Company: SAME
MSDS Serial Number: BXGGX

Ingredients/Identity Information

Proprietary: NO
Ingredient: CADMIUM (SARA 313) (CERCLA)
Ingredient Sequence Number: 01
NIOSH (RTECS) Number: EU9800000
CAS Number: 7440-43-9
OSHA PEL: 0.2 MG/M3 DUST; Z-2
ACGIH TLV: 0.01 MG/M3 DUST;9495

Physical/Chemical Characteristics

Appearance And Odor: SILVERY METALLIC GRANULES OR SHOT. ODORLESS.
Boiling Point: 1413F,767C
Melting Point: 610F,321C
Vapor Pressure (MM Hg/70 F): 394C
Specific Gravity: 1.04 (FP N)

Fire and Explosion Hazard Data

Flash Point: NOT APPLICABLE
Lower Explosive Limit: N/A
Upper Explosive Limit: N/A
Extinguishing Media: MEDIA SUITABLE FOR SURROUNDING FIRE (FP N). FIGHT SURROUNDING FIRE.
Special Fire Fighting Proc: USE NIOSH/MSHA APPROVED SCBA AND FULL PROTECTIVE EQUIPMENT (FP N).
Unusual Fire And Expl Hazrds: NOT COMBUSTIBLE. CADMIUM VAPOR MAY FORM IN GENERAL FIRE. AVOID INHALATION OF FUMES.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MANUFACTURER.
Materials To Avoid: NONE SPECIFIED BY MANUFACTURER.

Health Hazard Data

Precautions for Safe Handling and Use

Control Measures

Transportation Data

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 04MAY95
Label Date: 04MAY95
Label Status: G
Common Name: CADMIUM, 1147
Chronic Hazard: YES
Signal Word: CAUTION!
Acute Health Hazard-Slight: X
Contact Hazard-Slight: X
Fire Hazard-None: X
Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE: VAPOR HAZARDOUS IF INHALED. MUST BE HEATED STRONGLY TO PRODUCE VAPOR. INHALATION OF DUSTS OR VAPORS MAY LEAD TO PROBLEMS IN RESPIRATIORY TRACT KIDNEYS. SOLUBLE COMPOUNDS CONSIDERED MUCH MORE HAZARDOUS THAN METAL PIECES. CHRONIC: CANCER HAZARD. CONTAINS CADMIUM WHICH IS LISTED AS A GENITO-URINARY TRACT CARCINOGEN (FP N).
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: GFS CHEMICALS INC
Label P.O. Box: 245
Label City: POWELL
Label State: OH
Label Zip Code: 43065
Label Country: US
Label Emergency Number: 800-858-9682

G O CARLSON -- C 800 - NICKEL CHROMIUM IRON ALLOY
MATERIAL SAFETY DATA SHEET
NSN: 343900F027542
Manufacturer's CAGE: GOCAR
Part No. Indicator: A
Number/Trade Name: C 800

General Information

Item Name: NICKEL CHROMIUM IRON ALLOY
Company's Name: G O CARLSON INC
Company's Street: 350 MARSHALLTON RD
Company's City: THORNDALE
Company's State: PA
Company's Country: US
Company's Zip Code: 19372
Company's Emerg Ph #: 215-384-2800
Company's Info Ph #: 215-384-2800
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 03JUN92
Safety Data Review Date: 19MAY93
Preparer's Company: G O CARLSON INC
Preparer's St Or P. O. Box: 350 MARSHALLTON RD
Preparer's City: THORNDALE
Preparer's State: PA
Preparer's Zip Code: 19372
MSDS Serial Number: BQZSL

Ingredients/Identity Information

Proprietary: NO
Ingredient: IRON
Ingredient Sequence Number: 01
Percent: 50
NIOSH (RTECS) Number: NO4565500
CAS Number: 7439-89-6
OSHA PEL: 10 MG/CUM FUME
ACGIH TLV: 5 MG/CUM FUME

Proprietary: NO
Ingredient: CHROMIUM METAL, CHROMIUM, CHROME (SUSPECTED A1 HUMAN CARCINOGEN BY IARC, NTP & ACGIH)
Ingredient Sequence Number: 02
Percent: 19-23
NIOSH (RTECS) Number: GB4200000
CAS Number: 7440-47-3
OSHA PEL: 1 MG/CUM (CEILING)
ACGIH TLV: 0.5 MG/CUM

Proprietary: NO
Ingredient: NICKEL (SOLUBLE) (SUSPECTED HUMAN CARCINOGEN BY NTP, IARC, ACGIH) INTENDED CHANGE (IC)
Ingredient Sequence Number: 03
Percent: 30-35
NIOSH (RTECS) Number: QR5950000
CAS Number: 7440-02-0
OSHA PEL: 1 MG/CUM
ACGIH TLV: 0.05 MG/CUM IC
Other Recommended Limit: 1 MG/CUM

Proprietary: NO
Ingredient: MANGANESE, MN COMPOUNDS
Ingredient Sequence Number: 04
Percent: <1.5
NIOSH (RTECS) Number: OO9275000
CAS Number: 7439-96-5
OSHA PEL: (C) 5 MG/M3 DUST
ACGIH TLV: 5 MG/M3 DUST 9293

Proprietary: NO

Ingredient: COBALT
Ingredient Sequence Number: 05
Percent: <0.5
NIOSH (RTECS) Number: GF8750000
CAS Number: 7440-48-4
OSHA PEL: 0.1 MG/M3;AS CO
ACGIH TLV: 0.05 MG/M3;DUST 9293
Other Recommended Limit: 0.05 PPM

Physical/Chemical Characteristics

Appearance And Odor: ODORLESS SOLID W/METALLIC LUSTRE. AVAILABLE PLATES, DISCS, HEADS & SLABS.
Specific Gravity: 8-8.9

Fire and Explosion Hazard Data

Reactivity Data

Stability: YES
Hazardous Decomp Products: METAL FUMES & CERTAIN NOXIOUS GASES SUCH AS CO. ACID PICKLING MAY RESULT IN THE FORMATION OF HEXAVALENT CHROMIUM.
Hazardous Poly Occur: NO

Health Hazard Data

Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: NO
Health Haz Acute And Chronic: NO TOXIC EFFECTS EXPECTED FROM INERT FORM. EXPOSURE TO FUMES OR DUSTS GENERATED DURING HEATING, CUTTING, BRAZING OR WELDING MAY CAUSE ADVERSE HEALTH EFFECTS. INHALATION: IRRITATION OF NOSE & THROAT. EYES: IRRITATION. INGESTION: IRRITATION OF THE MOUTH & THROAT.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: SEE INGREDIENTS
Signs/Symptoms Of Overexp: INHALATION: PNEUMONITIS, SIDEROSIS. CNS INVOLVEMENT. INTERSTITIAL PNEUMONITIS & SENSITIZATION. NODULAR PULMONARY DISEASE. SKIN: DERMATITIS DUE TO SENSITIZATION.
Emergency/First Aid Proc: SKIN: WASH W/SOAP OR MILD DETERGENT & WATER FOR OBTAIN MEDICAL ATTENTION IN ALL CASES.

Precautions for Safe Handling and Use

Precautions-Handling/Storing: DURING WELDING, PRECAUTIONS SHOULD BE TAKEN FOR AIRBORNE CONTAMINANTS & NOXIOUS GASES FROM THE WELDING PROCESS OR COMPONENTS OF THE WELDING ROD.
Other Precautions: ARC & SPARKS GENERATED WHEN WELDING W/THIS PRODUCT COULD BE A SOURCE OF IGNITION FOR COMBUSTIBLE & FLAMMABLE MATERIALS.

Control Measures

Respiratory Protection: USE A PROPERLY FITTED NIOSH APPROVED DUST-FUME RESPIRATOR DURING WELDING OR BURNING.
Ventilation: RECOMMENDED TO KEEP BELOW TLV
Protective Gloves: RECOMMENDED
Eye Protection: SAFETY GLASSES OR GOGGLES
Work Hygienic Practices: REMOVE/LAUNDER CONTAMINATED CLOTHING BEFORE REUSE.
Suppl. Safety & Health Data: SOME CONSTITUENTS POSE MORE POTENTIAL HAZARDS THAN OTHERS, DEPENDING UPON THEIR INHERENT TOXICITY & CONCENTRATION. OF SPECIAL CONCERN ARE CHROMIUM, NICKEL & PERHAPS MANGANESE.

Transportation Data

Disposal Data

Label Data

Label Required: NO
Technical Review Date: 19MAY93
Label Date: 29APR93
Label Status: N
Label Name: G O CARLSON INC
Label Street: 350 MARSHALLTON RD
City: THORNDALE
State: PA
Label Zip Code: 19372
Label Country: US
Label Emergency Number: 215-384-2800

TARACORP INDUSTRIES EVANS METAL DIV -- LEAD (FABRICATIONS-FORMS);N-L 001 - WEIGHT, BALLAST
MATERIAL SAFETY DATA SHEET
NSN: 2040002746215
Manufacturer's CAGE: 27177
Part No. Indicator: A
Part Number/Trade Name: LEAD (FABRICATIONS/FORMS);N/L 001

General Information

Item Name: WEIGHT, BALLAST
Company's Name: TARACORP INDUSTRIES, EVANS METAL DIV
Company's Street: 740 LAMBERT DR NE
Company's City: ATLANTA
Company's State: GA
Company's Country: US
Company's Zip Code: 30324-4126
Company's Emerg Ph #: 404-875-5636 800-424-9300(CHEMTREC)
Company's Info Ph #: 404-875-5636
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 01MAR92
Safety Data Review Date: 13DEC96
Supply Item Manager: CX
MSDS Preparer's Name: UNKNOWN
MSDS Serial Number: BPYGW
Specification Number: MIL-W-20096
Spec Type, Grade, Class: NONE
Hazard Characteristic Code: T6
Unit Of Issue: EA
Unit Of Issue Container Qty: 50-60 LBS.
Type Of Container: UNKNOWN
Net Unit Weight: 50-60 LBS.

Ingredients/Identity Information

Proprietary: NO
Ingredient: LEAD (SARA III)
Ingredient Sequence Number: 01
Percent: >99.8
NIOSH (RTECS) Number: OF7525000
CAS Number: 7439-92-1
OSHA PEL: 0.05 MG/M3;1910.1025
ACGIH TLV: 0.15 MG/M3;DUST 9293
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: SOLID;SILVER METALLIC TO GRAY METALLIC METAL;NO ODOR.
Boiling Point: 3171F,1744C
Melting Point: 622F,328C
Specific Gravity: 11.34
Solubility In Water: INSOLUBLE
Corrosion Rate (IPY): UNKNOWN

Fire and Explosion Hazard Data

Flash Point: NONE
Extinguishing Media: USE MEDIA APPROPRIATE FOR SURROUNDING FIRE.
Special Fire Fighting Proc: USE A SELF-CONTAINED BREATHING APPARATUS AND FULL PROTECTIVE EQUIPMENT.
Unusual Fire And Expl Hazrds: NEVER MIX MOLTEN METAL WITH WATER;IT WILL EXPLODE.FIRE CONDITIONS MAY EVOLVE TOXIC FUMES.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MANUFACTURER.
Materials To Avoid: STRONG OXIDIZERS, HYDROGEN PEROXIDE, ACTIVE METALS(EG. SODIUM, POTASSIUM).AMMONIUM NITRATE(EXPLODES).
Hazardous Decomp Products: LEAD OXIDE FUMES ABOVE THE MELTING POINT.
Hazardous Poly Occur: NO

Health Hazard Data

LD50-LC50 Mixture: ORAL LD50 (RAT) IS 6789MG/KG
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: NO
Of Entry - Ingestion: YES
Haz Acute And Chronic: EXPOSURE TO SOLID FORM PRESENTS FEW HEALTH HAZARDS.NORMAL HANDLING OR PROCESSING OF THIS MATERIAL MAY RESULT IN THE GENERATION OF LEAD DUST AND/ OR FUMES. PROLONGED OVEREXPOSURE TO LEAD DUSTS OR FUMES CAN RESULT IN SYSTEMIC LEAD POISONING.CHRONIC:MAY DAMAGE BLOOD FORMING ORGANS,NERVOUS,KIDNEYS AND REPRODUCTIVE SYSTEMS.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: LEAD IS A CARCINOGEN WHICH HAS BEEN LISTED BY NTP,IARC AND EPA.LEAD IS TOXIC TO THE FETUS.
Signs/Symptoms Of Overexp: METALLIC TASTE,ANEMIA, INSOMNIA, WEAKNESS, CONSTIPATION, ABDOMINAL PAIN, GASTROINTESTINAL DISORDERS, JOINT & MUSCLE PAINS, & MUSCULAR WEAKNESS. SEVERE OVEREXPOSURE MAY LEAD TO CENTRAL NERVOUS SYSTEM DISORDERS, CHARACTERIZED BY DROWSINESS, STUPOR & ULTIMATELY DEATH. UNUSUAL CHRONIC TOXICITY-DEPRESSION OF BLOOD-FORMING.
Med Cond Aggravated By Exp: DISEASES OF THE SKIN,EYES,BLOOD FORMING ORGANS,KIDNEYS, NERVOUS & REPRODUCTIVE SYSTEM MAY BE AGGRIVATED BY EXPOSURE TO THIS PRODUCT.
Emergency/First Aid Proc: SKIN:REMOVE CONTAMINATED CLOTHING;WASH WITH SOAP AND WATER.EYES:FLUSH WITH WATER FOR 15 MINUTES.IF IRRITATION PERSISTS, GET MEDICAL ATTENTION.INHAL:REMOVE TO FRESH AIR.GIVE OXYGEN OR ARTIFICIAL RESPIRATION IF NEEDED.INGEST:GET PROMPT QUALIFIED MEDICAL ATTENTION.IF CONSCIOUS (AND NOT CONVULSING),GIVE WATER AND INDUCE VOMITING.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: VENTILATE AREA.USE PROPER PROTECTIVE AND RESPIRATORY EQUIPMENT.SWEEP OR VACUUM,USING A HEPA FILTER;DO NOT CREATE DUST;PLACE IN A DRY CONTAINER FOR DISPOSAL.CLEAN SPILL AREA WITH WATER.DO NOT CONTAMINATE WATERWAYS.
izing Agent: NOT APPLICABLE.
Disposal Method: RECYCLE WHERE POSSIBLE.DISPOSE OF IN ACCORDANCE WITH FEDERAL,STATE AND LOCAL REGULATIONS.CONSIDER RCRA CODE D008.
Precautions-Handling/Storing: STORE IN A COOL,DRY,WELL-VENTILATED PLACE. KEEP CONTAINER CLOSED WHEN NOT IN USE.KEEP AWAY FROM INCOMPATIBLE MATERIALS.
Other Precautions: **KEEP AWAY FROM FOOD** A PRE-EMPLOYMENT ACCESSMENT SHOULD BE PERFORMED IAW THE OSHA LEAD STANDARD 29CFR1910.1025.SPILLS SHOULD BE REPORTED TO THE NATIONAL RESPONSE CENTER AT 800-424-8802.

Control Measures

Respiratory Protection: WHERE ENVIRONMENTAL CONTROLS ARE LACKING OR IN ENCLOSED SPACES USE EITHER A SELF-CONTAINED BREATHING APPARATUS OR A NIOSH/ MSHA APPROVED RESPIRATOR FOR LEAD FUMES,DEPENDING ON THE AIRBORN CONCENTRATION.
Ventilation: USE GENERAL DILUTION VENTILATION.
Protective Gloves: IMPERVIOUS
Eye Protection: SAFETY GLASSES OR GOGGLES.
Other Protective Equipment: PROTECTIVE CLOTHING,AS NEEDED.PROVIDE A LOCAL EYE WASH STATION AND SAFETY SHOWER.
Work Hygienic Practices: WASH HANDS.SEPERATE WORK CLOTHES FROM STREET CLOTHES.LAUNDER WORK CLOTHES BEFORE REUSE.KEEP FOOD OUT OF THE WORK AREA.
Suppl. Safety & Health Data: NONE

Transportation Data

Trans Data Review Date: 96348
DOT PSN Code: ZZZ
DOT Proper Shipping Name: NOT REGULATED BY THIS MODE OF TRANSPORTATION
IATA PSN Code: ZZZ
IATA Proper Shipping Name: NOT REGULATED FOR THIS MODE OF TRANSPORTATION
IATA PSN Code: ZZZ
IATA Proper Shipping Name: NOT REGULATED BY THIS MODE OF TRANSPORTATION
AFI PSN Code: ZZZ
AFI Prop. Shipping Name: NOT REGULATED BY THIS MODE OF TRANSPORTATION

MMAC Code: NR
Additional Trans Data: CERCLA RQ IS 10 LBS, HOWEVER NO REPORTING IS REQUIRED IF SOLID PIECE IS GREATER THAN 100UM(0.004 INCHES) IN DIAMETER.

Disposal Data

Label Data

Label Required: NO
Technical Review Date: 13DEC96
MFR Label Number: UNKNOWN
Label Status: F
Common Name: LEAD (FABRICATIONS/FORMS)
Signal Word: WARNING!
Acute Health Hazard-Moderate: X
Contact Hazard-Slight: X
Fire Hazard-Slight: X
Reactivity Hazard-None: X
Special Hazard Precautions: NORMAL HANDLING OR PROCESSING OF THIS MATERIAL MAY RESULT IN THE GENERATION OF LEAD DUST AND/ OR FUMES. PROLONGED OVEREXPOSURE TO LEAD DUSTS OR FUMES CAN RESULT IN SYSTEMIC LEAD POISONING. CHRONIC:MAY DAMAGE BLOOD FORMING ORGANS,NERVOUS,KIDNEYS AND REPRODUCTIVE SYSTEMS. FIRST AID: SKIN:REMOVE CONTAMINATED CLOTHING;WASH WITH SOAP AND WATER.EYES:FLUSH WITH WATER FOR 15 MINUTES.IF IRRITATION PERSISTS,GET MEDICAL ATTENTION.INHAL:REMOVE TO FRESH AIR.GIVE OXYGEN OR ARTIFICIAL RESPIRATION IF NEEDED.INGEST:GET PROMPT QUALIFIED MEDICAL ATTENTION.IF CONSCIOUS (AND NOT CONVULSING),GIVE WATER AND INDUCE VOMITING.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: TARACORP INDUSTRIES, EVANS METAL DIV
Label Street: 740 LAMBERT DR NE
Label City: ATLANTA
Label State: GA
Label Zip Code: 30324-4126
Label Country: US
Label Emergency Number: 800-241-4590

JOHNSON MATTHEY CATALOG -- 13044 ARSENIC LUMP
MATERIAL SAFETY DATA SHEET
NSN: 685000F034390
Manufacturer's CAGE: 0JVJ1
Part No. Indicator: A
Number/Trade Name: 13044 ARSENIC LUMP

General Information

Company's Name: JOHNSON MATTHEY CATALOG CO
Company's Street: 30 BOND ST
Company's City: WARD HILL
Company's State: MA
Company's Country: US
Company's Zip Code: 01835-0747
Company's Emerg Ph #: 508-777-1970/508-521-6300
Company's Info Ph #: 508-777-1970/508-521-6300
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 11MAY94
Safety Data Review Date: 23JUN94
Preparer's Company: JOHNSON MATTHEY CATALOG CO
Preparer's St Or P. O. Box: 30 BOND ST
Preparer's City: WARD HILL
Preparer's State: MA
Preparer's Zip Code: 01835-0747
MSDS Serial Number: BTNMG

Ingredients/Identity Information

Proprietary: NO
Ingredient: ARSENIC, ARSENICALS (CONFIRMED HUMAN CARCINOGEN BY OSHA, NTP, IARC - GROUP 1) *94-2*
Ingredient Sequence Number: 01
Percent: 100
H (RTECS) Number: CG0525000
Number: 7440-38-2
HA PEL: 0.5 MG/CUM
ACGIH TLV: 0.2 MG/CUM

Physical/Chemical Characteristics

Appearance And Odor: STEEL-GRAY BRITTLE SOLID/NO ODOR
Boiling Point: 1135.4F
Melting Point: 1502.6F
Vapor Pressure (MM Hg/70 F): 1
Specific Gravity: 5.72
Evaporation Rate And Ref: (BU AC = 1): 0
Solubility In Water: INSOLUBLE
Percent Volatiles By Volume: 0

Fire and Explosion Hazard Data

Extinguishing Media: CO2, DRY CHEMICAL EXTINGUISHING AGENTS, DRY SAND/DRY GROUND DOLOMITE.
Special Fire Fighting Proc: WEAR NIOSH/MSHA APPROVED SCBA, FLAME & CHEMICAL RESISTANT PROTECTIVE CLOTHING. IF W/O RISK, REMOVE MATERIAL FROM FIRE AREA.
Unusual Fire And Expl Hazrds: SLIGHT EXPLOSION HAZARD IN THE FORM OF DUST WHEN EXPOSED TO FLAME. MODERATE FIRE HAZARD IN THE FORM OF DUST WHEN EXPOSED TO HEAT/FLAME/BY CHEMICAL REACTION.

Reactivity Data

Stability: YES
To Avoid (Stability): HEAT, FLAME, EXPOSURE TO AIR
als To Avoid: ACIDS, ACID FUMES, OXIDIZING AGENTS, HALOGENS, LADIUM, ZINC, PLATINUM, NITROGEN TRICHLORIDE, SILVER NITRATE (SUPP)
Hazardous Decomp Products: ARSENIC OXIDES
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): EXPOSURE TO AIR

Health Hazard Data

Route Of Entry - Inhalation: YES
Route Of Entry - Skin: NO
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: SKIN: MODERATE IRRITATION/SENSITIZATION. EYES: MODERATE IRRITATION. INHALATION: IRRITATION OF MUCOUS MEMBRANES/ RESPIRATORY TRACT/PHARYNGITIS. ARSENIC IS A NEUROTOXIN. POISONING MAY EFFECT THE HEART/GI SYSTEM/KIDNEYS & LIVER. CHRONIC-SKIN: ECZEMATOUS DERMATITIS/PIGMENTATION/HYPERKERATOSIS.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: SEE INGREDIENTS.
Signs/Symptoms Of Overexp: VOMITING, DIARRHEA, NAUSEA, IRRITATION, METALLIC TASTE, BLOODY NOSE, PERFORATION OF NASAL SEPTUM.
Emergency/First Aid Proc: SKIN: FLUSH W/PLENTY OF WATER. EYE: FLUSH W/ PLENTY OF WATER FOR 15 MINS. OBTAIN MEDICAL ATTENTION IN ALL CASES.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: WEAR FULL PROTECTIVE EQUIPMENT, COVER W/DRY SAND/VERMICULITE. MIX WELL & CAREFULLY TRANSFER TO A CONTAINER.
Waste Disposal Method: DISPOSE OF IAW/FEDERAL, STATE & LOCAL REGULATIONS.
UN1558
Precautions-Handling/Storing: KEEP CONTAINER TIGHTLY CLOSED. STORE IN COOL, DRY, WELL-VENTILATED AREA.
THIS PRODUCT CONTAINS A CHEMICAL KNOWN & CAUSE CANCER.

Control Measures

Respiratory Protection: REQUIRED
Ventilation: GLOVE BAG/BOX PREFERRED
Protective Gloves: RUBBER
Eye Protection: SAFETY GOGGLES W/FULL FACE SHIELD.
Other Protective Equipment: LAB COAT/APRON/FLAME & CHEMICAL RESISTANT COVERALLS/EYEWASH/SAFETY SHOWER.
Work Hygienic Practices: WASH THOROUGHLY AFTER USE. REMOVE/LAUNDER CONTAMINATED CLOTHING BEFORE REUSE
Suppl. Safety & Health Data: MAT TO AVOID CONT'D: ACETYLENES, CHLOROSYLAMINE, CHROMIUM VI OXIDE, SOLDIUM PEROXIDE, DIRUBIDIUM ACETYLIDE.

Transportation Data

Disposal Data

Label Data

Label Required: NO
Technical Review Date: 24JUN94
Label Date: 24JUN94
Label Status: N
Special Hazard Precautions: POSSIBLE LUNG IRRITAITON FROM SILICA. LONG QUARTZ.
Label Name: JOHNSON MATTHEY CATALOG CO
Label Street: 30 BOND ST
Label City: WARD HILL
Label State: MA
Label Zip Code: 01835-0747
Label Country: US
Label Emergency Number: 508-777-1970/508-521-6300

DUPONT E I AUTOMOTIVE PRODS D -- 2319S LACQUER THINNERS & CLEANING SOLVENTS - PAINTS
MATERIAL SAFETY DATA SHEET
NSN: 801000F049331
Manufacturer's CAGE: 90227
Part No. Indicator: A
P[illegible] Number/Trade Name: 2319S LACQUER THINNERS & CLEANING SOLVENTS

General Information

Item Name: PAINTS
Company's Name: DUPONT E I DE NEMOURS & CO INC AUTOMOTIVE PRODS D
Company's Street: BRANDYWINE BLDG 3332 1007 MARKET ST
Company's City: WILMINGTON
Company's State: DE
Company's Country: US
Company's Zip Code: 19898-5000
Company's Emerg Ph #: 800-441-3637/800-441-7515
Company's Info Ph #: 800-441-7515/800-441-3637
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SE
Date MSDS Prepared: 01JAN95
Safety Data Review Date: 09AUG96
MSDS Preparer's Name: T R LOUER
Preparer's Company: DUPONT E I DE NEMOURS & CO INC AUTOMOTIV
Preparer's St Or P. O. Box: BRANDYWINE BLDG 3332 1007 MARKET ST
Preparer's City: WILMINGTON
Preparer's State: DE
Preparer's Zip Code: 19898-5000
MSDS Serial Number: BZVRG

Ingredients/Identity Information

Proprietary: NO
Ingredient: ACETIC ACID (GLACIAL), ETHANOIC ACID *96-2*
Ingredient Sequence Number: 01
N[illegible] (RTECS) Number: AF1225000
([illegible]nber: 64-19-7
OS[illegible]PEL: 25 MG/CUM
ACGIH TLV: 25 MG/CUM

Proprietary: NO
Ingredient: ISOPROPANOL (ISOPROPYL ALCOHOL), 2-PROPANOL, DIMETHYL CARBINOL (IARC CANCER REVIEW GROUP 3) *96-2*
Ingredient Sequence Number: 02
NIOSH (RTECS) Number: NT8050000
CAS Number: 67-63-0
OSHA PEL: 400 PPM
ACGIH TLV: 400 PPM
Other Recommended Limit: 400 PPM

Proprietary: NO
Ingredient: WATER
Ingredient Sequence Number: 03
NIOSH (RTECS) Number: ZC0110000
CAS Number: 7732-18-5

Proprietary: NO
Ingredient: VOLATILE ORGANIC CONTENT: 6.6 LBS/GAL
Ingredient Sequence Number: 04
NIOSH (RTECS) Number: 9999999VO

Physical/Chemical Characteristics

Boiling Point: 129-509F
Vapor Density (Air=1): >1
Evaporation Rate And Ref: LESS THAN ETHER
[illegible] In Water: MISCIBLE
[illegible]olatiles By Volume: 93-100

Fire and Explosion Hazard Data

Flash Point: <73F
Flash Point Method: CC
Lower Explosive Limit: 0.8
Upper Explosive Limit: 36.5
Extinguishing Media: WATER SPRAY, FOAM, CO2, DRY CHEMICAL
Special Fire Fighting Proc: WEAR FULL PROTECTIVE EQUIPMENT W/SELF CONTAINED BREATHING APPARATUS. WATER FROM FOG NOZZLES MAY BE USED TO COOL CLOSED CONTAINERS TO PREVENT PRESSURE BUILDUP.
Unusual Fire And Expl Hazrds: WHEN HEATED >FLASH POINT, EMITS FLAMMABLE VAPORS WHICH, WHEN MIXED W/AIR, CAN BURN/BE EXPLOSIVE. FINE MISTS/SPRAYS MAY BE FLAMMABLE AT TEMPS 120F & OTHER IGNITION SOURCES
Materials To Avoid: NONE
Hazardous Decomp Products: CO, CO2, SMOKE
Hazardous Poly Occur: NO

Health Hazard Data

Route Of Entry - Inhalation: YES
Route Of Entry - Skin: NO
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: INGESTION: MAY CAUSE GASTROINTESTINAL DISTRESS. INHALATION: NOSE & THROAT IRRITANT. OVEREXPOSURE TO SOLVENTS MAY LEAD TO PERMANENT BRAIN & NERVOUS SYSTEM DAMAGE. EYES: MAY CAUSE IRRITATION/BURNING. SKIN: IRRITATION W/DISCOMFORT, DERMATITIS.
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: NONE
Signs/Symptoms Of Overexp: GI DISTRESS, IRRITATION, EYE WATERING, HEADACHES, NAUSEA, DIZZINESS, LOSS OF COORDINATION, BURNING
IMMEDIATELY FLUSH W/PLENTY OF WATER FOR 15 MINS. SKIN: WASH W/SOAP & WATER. OBTAIN MEDICAL ATTENTION IN ALL CASES.

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: VENTILATE AREA. REMOVE SOURCES OF IGNITION. PREVENT SKIN CONTACT & BREATHING OF VAPOR. WEAR A PROPERLY FITTED VAPOR/PARTICULATE RESPIRATOR (NIOSH/MSHA TC-23C). CONFINE & REMOVE W/INERT ABSORBENT.
Waste Disposal Method: DON'T ALLOW MATERIAL TO CONTAMINATE GROUND WATER SYSTEMS. INCINERATE ABSORBED MATERIAL IAW/LOCAL, STATE & FEDERAL REGULATIONS. DON'T INCINERATE IN CLOSED CONTAINERS.
Precautions-Handling/Storing: DON'T STORE >120F. CLOSE CONTAINER AFTER EACH USE. GROUND CONTAINERS WHEN POURING. OBSERVE LABEL PRECAUTIONS. KEEP AWAY FROM HEAT, SPARKS & FLAME.
Other Precautions: DON'T SAND FLAME CUT, BRAZE/WELD DRY COATING W/O A NIOSH/MSHA APPROVED RESPIRATOR/APPROPRIATE VENTILATION. DON'T PERMIT ANYONE W/O PROTECTION IN THE PAINTING AREA. PREVENT SKIN CONTACT & DON'T BREATHE VAPORS/MISTS.

Control Measures

Respiratory Protection: WEAR A PROPERLY FITTED VAPOR/PARTICULATE RESPIRATOR APPROVED BY NIOSH/MSHA (TC-23C) FOR USE W/PAINTS DURING APPLICATION & UNTIL ALL VAPORS & SPRAY MISTS ARE EXHAUSTED. IN CONFINED SPACES, WEAR A POSITVE PRESSURE, SUPPLIED AIR RESPIRATOR.
Ventilation: SUFFICIENT VENTILATION IN VOLUME & PATTERN TO KEEP CONTAMINANTS <APPLICABLE OSHA REQUIREMENTS.
Protective Gloves: NEOPRENE
Eye Protection: SPLASH GUARDS/SIDE SHIELDS
Other Protective Equipment: COVERALLS
Work Hygienic Practices: WASH THOROUGHLY AFTER HANDLING & BEFORE EATING/SMOKING.

Transportation Data

Disposal Data

Label Data

Label Required: YES
Label Status: G
Common Name: 2319S LACQUER THINNERS & CLEANING SOLVENTS
Special Hazard Precautions: INGESTION: MAY CAUSE GASTROINTESTINAL DISTRESS. INHALATION: NOSE & THROAT IRRITANT. OVEREXPOSURE TO SOLVENTS MAY LEAD TO PERMANENT BRAIN & NERVOUS SYSTEM DAMAGE. EYES: MAY CAUSE
[illegible]ATION/BURNING. SKIN: IRRITATION W/DISCOMFORT, DERMATITIS. GI DISTRESS,
[illegible]TION, EYE WATERING, HEADACHES, NAUSEA, DIZZINESS, LOSS OF
[illegible]RDINATION, BURNING
Label Name: DUPONT E I DE NEMOURS & CO INC AUTOMOTIVE PRODS D
Label Street: BRANDYWINE BLDG 3332 1007 MARKET ST
Label City: WILMINGTON
Label State: DE
Label Zip Code: 19898-5000
Label Country: US
Label Emergency Number: 800-441-3637/800-441-7515

JEM SALES -- F-176(1,1,1-TRICHLOROETHANE) - TRICHLOROETHANE,TECHNICAL
MATERIAL SAFETY DATA SHEET
NSN: 6810006640387
Manufacturer's CAGE: 0AZD7
Part No. Indicator: B
Part Number/Trade Name: F-176(1,1,1-TRICHLOROETHANE)

General Information

Item Name: TRICHLOROETHANE,TECHNICAL
Company's Name: JEM SALES,INC
Company's Street: 430 LAVENDER DRIVE
Company's City: ROME
Company's State: GA
Company's Country: US
Company's Zip Code: 30165-7762
Company's Emerg Ph #: 800-424-9300(CHEMTREC)
Company's Info Ph #: 706-232-1709/FAX 706-802-1175
Record No. For Safety Entry: 003
Tot Safety Entries This Stk#: 017
Status: SE
Date MSDS Prepared: 02APR96
Safety Data Review Date: 28APR97
Supply Item Manager: GSA
MSDS Preparer's Name: GEORGE KORDARRES
MSDS Serial Number: CDQLV
Spec Type, Grade, Class: TYPE I
Hazard Characteristic Code: T4
Unit Of Issue: GL
Unit Of Issue Container Qty: 1 GALLON
Type Of Container: CAN
Net Unit Weight: 11.0 LBS

Ingredients/Identity Information

Proprietary: NO
Ingredient: METHYL CHLOROFORM (1,1,1-TRICHLOROEHANE) (SARA 313) (CERCLA)
Ingredient Sequence Number: 01
Percent: >95
NIOSH (RTECS) Number: KJ2975000
CAS Number: 71-55-6
OSHA PEL: 350 PPM
ACGIH TLV: 350 PPM/450STEL;9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: TERT-BUTYL ALCOHOL (SARA 313)
Ingredient Sequence Number: 02
Percent: 1.9
NIOSH (RTECS) Number: EO1925000
CAS Number: 75-65-0
OSHA PEL: 100 PPM
ACGIH TLV: 100 PPM, A4; 9596
Other Recommended Limit: NONE RECOMMENDED

Proprietary: NO
Ingredient: VOLITILE ORGANIC CONTENT AS LISTED ON MSDS, 11.0 LBS/GAL; 1319 G/L.
Ingredient Sequence Number: 03
Percent: NA
NIOSH (RTECS) Number: 9999999VC
OSHA PEL: NOT ESTABLISHED
ACGIH TLV: NOT ESTABLISHED
Other Recommended Limit: NONE RECOMMENDED

Physical/Chemical Characteristics

Appearance And Odor: LIQUID, CLEAR, PARTICLE FREE LIQUID W/SWEET AROMA.
Boiling Point: 170F,77C
Melting Point: -35F,-37C
Vapor Pressure (MM Hg/70 F): 100 @20C
Vapor Density (Air=1): 4.6

Specific Gravity: 1.319
Decomposition Temperature: NP
Evaporation Rate And Ref: 35, ETHER=1.
Solubility In Water: 0.07G/100G @25C.
Percent Volatiles By Volume: 100
Viscosity: 0.86CP @20C
[illegible]A
[illegible]on Rate (IPY): NA
[illegible]gnition Temperature: 458C

Fire and Explosion Hazard Data

Flash Point: DOES NOT FLASH
Lower Explosive Limit: 7.0
Upper Explosive Limit: 15
Extinguishing Media: NOT APPLICABLE. USE WATER,FOG,FOAM,DRY CHEM OR CO2 FOR SURROUNDING FIRE.USE WATERSPRAY TO COOL FIRE-EXPO CNTNRS/EQPMT.
Special Fire Fighting Proc: DON'T RELEASE RUNOFF FROM FIRE CNTRL METHODS TO SEWERS/WATERWAYS.BECAUSE FIRE MAY PROD TOXIC THERM DEOCMPO PROD WEAR SCBA W/FULL FACEPIECE OPERATED IN (SUPPLEM)
Unusual Fire And Expl Hazrds: NON-FLAMM.NEVER USE WELD/CUT TORCH ON/NEAR DR(EVEN EMPTY)-PROD(EVEN RESIDUE)CAN IGN EXPLO.VAP CONC IN CONFINED &/OR POORLY VENTI AREA CAN BE IGN UPON (SUPPLEME)

Reactivity Data

Stability: YES
Cond To Avoid (Stability): OPEN FLAMES, SPARKS, HIGH TEMPERATURES, IGNITION SOURCES.
Materials To Avoid: WATER,REACTIVE METALS SUCH AS ALUMINUM/MAGNESIUM, STRONG OXIDIZING AGENTS, STRONG ALKALIES.
Hazardous Decomp Products: HAZ COMBUST PROD:HYDROGEN CHLORIDE, DICHLOROACETYLENE,PHOSGENE,CHLORINE GAS.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NONE SPECIFIED BY MANUFACTURER.

Health Hazard Data

[illegible]C50 Mixture: NONE SPECIFIED BY MANUFACTURER.
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: NO
Health Haz Acute And Chronic: DEFAT SKIN CAUSING IRRIT,REDNESS,DRYNESS, SCALING.EYE:IRRIT,MILD CONJUNCTIVITIS.VAP INHAL:HEAD,DIZZ,EQUILIBRIUM DISTURBANCES,HIGH CONC LEAD TO CNS DEPRESS,UNCONSC,COMA.MILD LIVE/KIDNEY DYSFUNCTION AFT CNS DEPRESS RECOVERY.INGEST:UNLIKELY IF SO NAU,VOMIT,DIARR, POSSIBLE ESOPHAGEAL BURNS.CHRONIC EFFECTS:NONE REPORTED.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: YES
Explanation Carcinogenicity: PER MSDS:IARC/NTP/OSHA LIST COMPONENT 1,1,1-TRICHLOROETHANE AS SUSPECTED CARCINOGEN.
Signs/Symptoms Of Overexp: INHAL:LIGHTHEADNEDNESS,IMPAIRED COORDINATION,DIZZINESS,DROWSINESS.EYE:BLURNING,STINGING,WATERING, CONJUNCTIVAL INFLAMM,POSSIBLE CORNEAL INJURY.SKIN:MILD IRRIT, DRYNESS(DEFATTING EFFECT),COLD FEELING.INGEST:DEPRESSION OF CNS,THROAT IRRIT,DIZZ,DROWSINESS,UNCONSC,DEATH.
Med Cond Aggravated By Exp: NONE REPORTED. TARGET ORGANS:SKIN,EYE, CENTRAL NERVOUS SYSTEM(CNS),CARDIOVASCULAR(CVS)SYSTEM.
Emergency/First Aid Proc: INHAL:REMOVE TO FRESH AIR.BREATH DIFF ADMIN OXY.EYE:DON'T ALLOW TO RUB OR KEEP TIGHTLY SHUT.FOUSH W/LG AMTS OF WTER. CONSULT DR.SKIN:QUICKLY REMOVE CONTAMIN CLOTH.RINSE W/LG AMTS OF WATER.WASH W/SOAP/WATER.RED/BLISTED SKIN CONSULT DR.INGEST:DO NOT INDUCE VOMIT.GIVE 1 OR 2 GLASSES OF WATER TO DRINK.NEVER GIVE ANYTHING BY MOUTH IF UNCONSC. CONTACT POIS CNTRL CNTR/MED PERSON.DR:AVOID USE OF (SUPPLEMEN)

Precautions for Safe Handling and Use

[illegible]Matl Released/Spill: NOTIFY SAF PERSON,EVACUATE ALL UNNECESSARY P[illegible]ONS,GET PPE.SM:ABSORB ON PAPER/VERMI/ABSORBENT MATL.TRANSFER TO EXHAU H[illegible]D/OUTSIDE.LG:STOP SPILL.DIKE,PUMP TO SALVAGE TK.USE ABSORBENT MATL FOR REMAINING LIQ.DON'T RELEAASE TO SEWER/WATERWAY.FOLLOW OSHA.
Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.

Waste Disposal Method: CONTACT SUPPLIER/LICD CONTRACTOR FOR DETAILED RECOMMENDATIONS.ANY DISPO MUST BE IN COMPLIANCE W/FED/STATE/LOC REGS.EMPTY CNTNR RETAIN PROD RESIDUE.DON'T DISTRIBUTE/MAKE AVAI/FURNISH/REUSE EMPTY CNTR EXCEPT FOR STORAGE/SHIP ORG PROD.REMOVE (OTH PRECA)
Precautions-Handling/Storing: CONSIDER PRE MED EXAMS OF EXPO WORKERS THAT EMPHASIZE CNS/CVS/LIVER/SKIN.INFO WORKER OF PROD HAZ & OSHA 29CFR1910.146. STORE:COOL DRY WELL VENTI AREA.
Other Precautions: DISPO:RESIDUE FRM CNTNR,PUNCT/DESTR EMPTY CNTNR BEF DISPO.OBSERVE ALL HAZ PRECAU.KEEP AWAY FRM HEAT/ETC.DON'T WELD/CUT TORCH ON/NEAR CNTNR.WASTE#:U226 FOR PROD;F001 FOR DEGRE/SLUD;F002 FOR STILL BOTTOM.RQ:1000LBS.SARA TOXI CHEM.AIR CONTAMIN

Control Measures

Respiratory Protection: GET PROF ADVICE PRIOT TO RESP SELECTION/USE.FOLLOW OSHA 29CFR1910.134.WEAR NIOSH/MSHA RESP.BASE SELECTION SUITABGILITY FOR PROT FOR CONDITONS,LEVEL OF CONTAIN,SUFFI OXY.EMER/NONRT OPERATIONS WEAR SCBA.AIRPURIF RESP NO PROTECT IN OXY DEFFIC.
Ventilation: PROVIDE GEN/LOC EXHAU VENTI(PREFER)SYS-MAINTAIN AIRBORNE CON BEL OSHA PELS.PROVIDE +VENTI SYS FOR ENTIRE ENTRY PERIOD.
Protective Gloves: CHEM PROTECTIVE GLOVES.
Eye Protection: PROT EYEGLASSES,CHEM SAF GOGG,FACE PROT.
Other Protective Equipment: CONTACT LENSES NOT EYE PROT DEVICES.CHEM PROT BOOTS/APRONS/GAUNTLETS.EMERG EYEWASH STATIONS,SAF/QUICK SHOWER,WASH FACILI
Work Hygienic Practices: SEPARATE CONTAM WORKCLOTH FRM STREETCLOTH.WASH CONTAM CLOTH BEF REUSE;REMOVE PROD FRM SHOE.CLEAN PPE.FOLLOW GOOD HYG PRA
Suppl. Safety & Health Data: FIREFIGHT:PRESSURE-DEMAND OR POSITIVE-PRESSURE MODE. FIRE/EXPLO:CONTACT W/HIGH ENERGY SPARK/FLAME/IGN SOURCE.VAP HEAVIER THAN AIR-COLLECT IN LOW AREAS. 1STAID:OF ADRENALIN OR HEART STIMULATING DRUGS WHEN OVERCOME BY PROD.BECAUSE RAPID ABSORP THRU LUNGS IF ASPIRATED,SYS EFFECTS OCCUR,DR SHOULD DECIDED IF VOMIT OR NOT.

Transportation Data

Trans Data Review Date: 97118
DOT PSN Code: OQD
DOT Proper Shipping Name: 1,1,1- TRICHLOROETHANE *
DOT Class: 6.1
DOT ID Number: UN2831
DOT Pack Group: III
DOT Label: KEEP AWAY FROM FOOD
IMO PSN Code: OVK
IMO Proper Shipping Name: 1,1,1- TRICHLOROETHANE *
IMO Regulations Page Number: 6272-1
IMO UN Number: 2831
IMO UN Class: 6.1
IMO Subsidiary Risk Label: -
IATA PSN Code: YLY
IATA UN ID Number: 2831
IATA Proper Shipping Name: 1,1,1- TRICHLOROETHANE *
IATA UN Class: 6.1
IATA Label: TOXIC
AFI PSN Code: YLY
AFI Prop. Shipping Name: 1,1,1- TRICHLOROETHANE *
AFI Class: 6.1
AFI ID Number: UN2831
AFI Pack Group: III
AFI Special Prov: N36
AFI Basic Pac Ref: A10.5
Additional Trans Data: PER MSDS:DOT TRANSPO INFO:SHIPPING NAME:1,1, 1-TRICHLOROETHANE, 6.1, UN 2831, PKG III, LABEL:KEEP AWAY FROM FOOD.

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 28APR97
Label Status: F
Common Name: F-176(1,1,1-TRICHLOROETHANE)
Chronic Hazard: NO
Signal Word: DANGER!

Acute Health Hazard-Severe: X
Contact Hazard-Slight: X
Fire Hazard-None: X
Reactivity Hazard-None: X
Special Hazard Precautions: DEFAT/IRRIT SKIN,RED,DRY,SCALING.EYE:IRRIT,
MILD CONJUNC.VAP INHAL:HEAD,DIZZ,EQUILIBRIUM DISTURB,HI CONC CNS DEPRESS,
LIKELY IF SO NAU,VOMIT,DIARR,ESOPHAGEAL BURNS.TARGET ORGANS:CNS/CVS/
R/SKIN.1STAID:INHAL:REMOVE TO FRESH AIR.BREATH DIFF ADMIN OXY.EYE:DON'T
QKLY REMOVE CONTAMIN CLOTH.RINSE W/LG AMTS OF WATER.WASH W/SOAP/WATER.
RED/BLISTED SKIN CONSULT DR.INGEST:DO NOT INDUCE VOMIT.GIVE 1 OR 2 GLASSES
OF WATER TO DRINK.NEVER GIVE ANYTHING BY MOUTH IF UNCONSC.CONTACT POIS
CNTRL CNTR/MED PERSON.D
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: JEM SALES,INC
Label Street: 430 LAVENDER DRIVE
Label City: ROME
Label State: GA
Label Zip Code: 30165-7762
Label Country: US
Label Emergency Number: 800-424-9300(CHEMTREC)

CHEM SERVICE – TRICHLOROETHENE, 0-664
MATERIAL SAFETY DATA SHEET
NSN: 681000N054678
Manufacturer's CAGE: 8Y898
Part No. Indicator: A
Part Number/Trade Name: TRICHLOROETHENE, 0-664

General Information

Company's Name: CHEM SERVICE INC
Company's P. O. Box: 3108
Company's City: WEST CHESTER
Company's State: PA
Company's Country: US
Company's Zip Code: 19381
Company's Emerg Ph #: 215-692-3026
Company's Info Ph #: 215-692-3026
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SMJ
Date MSDS Prepared: 07JAN93
Safety Data Review Date: 03NOV94
MSDS Serial Number: BVYRM
Hazard Characteristic Code: NK

Ingredients/Identity Information

Proprietary: NO
Ingredient: ETHYLENE, TRICHLORO-; (TRICHLOROETHYLENE) (SARA III)
Ingredient Sequence Number: 01
NIOSH (RTECS) Number: KX4550000
CAS Number: 79-01-6
OSHA PEL: 100 PPM
ACGIH TLV: 50 PPM;100 PPM STEL

Proprietary: NO
Ingredient: SUPP DATA: BRTHG ADMIN ARTF RESPS.IF PATIENT IS IN CARD ARREST ADMIN CPR. CONTINUE LIFE SUPPORTING MEASURES UNTIL(ING 3)
Ingredient Sequence Number: 02
NIOSH (RTECS) Number: 9999999ZZ
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: ING 2: MEDICAL ASSISTANCE HAS ARRIVED. INGESTION: CALL MD IMMEDIATELY (FP N).
Ingredient Sequence Number: 03
NIOSH (RTECS) Number: 9999999ZZ
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: EYE PROTECTION: FULL LENGTH FACESHIELD (FP N).
Ingredient Sequence Number: 04
NIOSH (RTECS) Number: 9999999ZZ
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Physical/Chemical Characteristics

Appearance And Odor: COLORLESS LIQUID.
Boiling Point: 189F,87C
Melting Point: -125F,-87C
Vapor Pressure (MM Hg/70 F): 58 @ 20C
Specific Gravity: 1.462
Solubility In Water: INSOLUBLE

Fire and Explosion Hazard Data

Flash Point: NON-FLAMMABLE
Lower Explosive Limit: 11%
Upper Explosive Limit: 41%

Extinguishing Media: CARBON DIOXIDE, DRY CHEMICAL POWDER OR SPRAY.
Special Fire Fighting Proc: WEAR NIOSH/MSHA APPROVED PRESSURE DEMAND SCBA AND FULL PROTECTIVE EQUIPMENT (FP N).
Unusual Fire And Expl Hazrds: THERMAL DECOMPOSITION PRODUCTS MAY INCLUDE HCL AND PHOSGENE (FP N).

Reactivity Data

[redacted]bility: YES
Cond To Avoid (Stability): NONE SPECIFIED BY MANUFACTURER.
Materials To Avoid: STRONG BASES, STRONG OXIDIZING AGENTS.
Hazardous Decomp Products: DECOMPOSITION LIBERATES TOXIC FUMES. DECOMPOSITION PRODUCTS ARE CORROSIVE. HCL, PHOSGENE (FP N).
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT RELEVANT.

Health Hazard Data

LD50-LC50 Mixture: LD50 (ORAL,RAT): 4920 MG/KG.
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: CONT LENSES SHOULD NOT BE WORN IN LAB. ALL CHEMS SHOULD BE CONSIDERED HAZ-AVOID DIRECT PHYS CONT! SUSPECTED CARCIN-MAY PRDCE CANCER. MAY BE HARMFUL IF ABSORB THRU SKIN. MAY BE HARMFUL IF INHALED. MAY BE HARMFUL IF SWALLOWED. LACHRYMATOR-CAUSES SEV EYE IRRIT. VAPS &/OR DIRECT EYE CONT CAN CAUSE SEV EYE (EFTS OF OVEREXP)
Carcinogenicity - NTP: NO
Carcinogenicity - IARC: NO
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: NOT RELEVANT.
Signs/Symptoms Of Overexp: HLTH HAZ: BURNS. CAN CAUSE EYE IRRIT. CAN CAUSE SKIN IRRIT. CAN CAUSE SKIN BURNS. CAN CAUSE SEV SKIN BURNS. EXPOS CAN CAUSE LIVER DMG. EXPOS CAN CAUSE KIDNEY DMG. CAN CAUSE GI DISTURB. CAN BE IRRIT TO MUC MEMBS. PRLNGD EXPOS MAY CAUSE NAUS/HDCH/DIZZ &/OR EYE DMG.CAN CAUSE SENSIT BY SKIN CONT. CHLOROCARBON MATLS(SUPDAT)
Med Cond Aggravated By Exp: NONE SPECIFIED BY MANUFACTURER.
[redacted]ency/First Aid Proc: AN ANTIDOTE IS SUBSTANCE INTENDED TO COUNTERACT [redacted]OF POIS. IT SHOULD BE ADMIN ONLY BY PHYS/TRAINED EMER PERS. MED ADVICE CAN BE OBTAINED FROM POIS CNTRL CNTR. EYE: FLUSH CONTINUOUSLY W/ WATER FOR AT LST 15-20 MINS. SKIN: FLUSH W/WATER FOR 15-20 MINS. IF NOT BURNS HAVE OCCURED-USE SOAP & WATER TO CLEANSE SKIN. INHAL: REMOVE PATIENT TO FRESH AIR. ADMIN OXYGEN IF PATIENT IS HAVING DFCLTY (SUPDAT)

Precautions for Safe Handling and Use

Steps If Matl Released/Spill: EVACUATE AREA. WEAR APPROPRIATE OSHA REGULATED EQUIPMENT. VENTILATE AREA. ABSORB ON VERMICULITE OR SIMILAR MATERIAL. SWEEP UP AND PLACE IN AN APPROPRIATE CONTAINER. HOLD FOR DISPOSAL. WASH CONTAMINATED SURFACES TO REMOVE ANY RESIDUES.
Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.
Waste Disposal Method: BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER. DISPOSE OF IN ACCORDANCE WITH FEDERAL, STATE, AND LOCAL REGULATIONS (FP N).
Precautions-Handling/Storing: AVOID CONTACT WITH SKIN, EYES AND CLOTHING. KEEP TIGHTLY CLOSED IN COOL DRY PLACE. STORE ONLY WITH COMPATIBLE CHEMICALS.
Other Precautions: NO SMOKING IN AREA OF USE. DO NOT USE IN GENERAL VICINITY OF ARC WELDING, OPEN FLAMES OR HOT SURFACES. HEAT AND/OR UV RADIATION MAY CAUSE FORMATION OF HCL AND/OR PHOSGENE (FP N).

Control Measures

Respiratory Protection: WEAR NIOSH/MSHA APPROVED RESPIRATOR APPROPRIATE FOR EXPOSURE OF CONCERN (FP N).
Ventilation: CHEMICAL SHOULD BE HANDLED ONLY IN HOOD.
Protective Gloves: IMPERVIOUS GLOVES (FP N).
[redacted]rotection: ANSI APPRVD CHEM WORKERS GOGG & (ING 4)
[redacted] Protective Equipment: USE APPROPRIATE OSHA/MSHA APPROVED SAFETY [redacted]PMENT.EMER EYEWASH & DELUGE SHOWER WHICH MEET ANSI DESIGN STANDARDS(FP N).
Work Hygienic Practices: NONE SPECIFIED BY MANUFACTURER.
Suppl. Safety & Health Data: EFTS OF OVEREXP: HAVE PRDCED SENSIT OF

MYOCARDIUM TO EPINEPHRINE IN LAB ANIMALS & COULD HAVE SIMILAR EFT IN HUMANS. ADRENOMIMETICS (E.G., EPINEPHRINE) MAY BE CONTRAINDICATED EXCEPT FOR LIFE-SUSTAINING USES IN HUMANS ACUTELY/CHRONICALLY EXPOS TO CHLOROCARBONS (FP N). FIRST AID PROC: BRTHG. IF PATIENT HAS STOPPED (ING 2)

Transportation Data

Disposal Data

Label Data

Label Required: YES
Technical Review Date: 03NOV94
Label Date: 26OCT94
Label Status: G
Common Name: TRICHLOROETHENE, 0-664
Chronic Hazard: YES
Signal Word: DANGER!
Acute Health Hazard-Moderate: X
Contact Hazard-Severe: X
Fire Hazard-None: X
Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE: INHALATION OF VAPORS MAY CONTRIBUTE TO THE OCCURRENCE OF IRREGULAR HEARTBEAT (FP N). MAY BE HARMFUL IF ABSORB THRU SEVERE BURNS/IRRITATION MAY CAUSE LIVER/KIDNEY DAMAGE, GASTROINTESTINAL DISTURBANCE. MAY CAUSE MUCOUS MEMBRANE IRRITATION. CHRONIC: NAUSEA, HEADACHE, DIZZINESS AND/OR EYE DAMAGE.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: CHEM SERVICE INC
Label P.O. Box: 3108
Label City: WEST CHESTER
Label State: PA
Label Zip Code: 19381
Label Country: US
Label Emergency Number: 215-692-3026

ENVIRONMENTAL RESOURCE ASSOCIATES -- PCB'S IN OIL, PT51
MATERIAL SAFETY DATA SHEET
NSN: 666500N072173
Manufacturer's CAGE: 1R664
Part No. Indicator: A
Number/Trade Name: PCB'S IN OIL, PT51

General Information

Company's Name: ENVIRONMENTAL RESOURCE ASSOCIATES
Company's Street: 5540 MARSHALL ST
Company's City: ARVADA
Company's State: CO
Company's Country: US
Company's Zip Code: 80002
Company's Emerg Ph #: 303-431-8454
Company's Info Ph #: 303-431-8454
Record No. For Safety Entry: 001
Tot Safety Entries This Stk#: 001
Status: SMJ
Date MSDS Prepared: 09JAN96
Safety Data Review Date: 15AUG96
MSDS Serial Number: CBPZQ

Ingredients/Identity Information

Proprietary: NO
Ingredient: AROCHLOR (ONLY ONE OF THE FOLLOWING AROCHLORS (INGS 2 - 10) WILL BE PRESENT IN EACH SAMPLE)
Ingredient Sequence Number: 01
NIOSH (RTECS) Number: 9999999ZZ
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1016) (SARA 313) (CERCLA)
Ingredient Sequence Number: 02
Percent: <0.05
NIOSH (RTECS) Number: TQ1351000
CAS Number: 12674-11-2
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1221); (MFR CAS #11104-16-2)
Ingredient Sequence Number: 03
Percent: <0.05
NIOSH (RTECS) Number: TQ1352000
CAS Number: 11104-28-2
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1232) (SARA 313) (CERCLA)
Ingredient Sequence Number: 04
Percent: <0.05
NIOSH (RTECS) Number: TQ1354000
CAS Number: 11141-16-5
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1242) (SARA 313) (CERCLA)
Ingredient Sequence Number: 05
Percent: <0.05
NIOSH (RTECS) Number: TQ1356000
CAS Number: 53469-21-9
OSHA PEL: 1 MG/M3, S
ACGIH TLV: 1 MG/M3, S

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1248) (SARA 313) (CERCLA)

Ingredient Sequence Number: 06
Percent: <0.05
NIOSH (RTECS) Number: TQ1358000
CAS Number: 12672-29-6
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1254) (SARA 313) (CERCLA)
Ingredient Sequence Number: 07
Percent: <0.05
NIOSH (RTECS) Number: TQ1360000
CAS Number: 11097-69-1
OSHA PEL: 0.5 MG/M3, S
ACGIH TLV: 0.5 MG/M3, S

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1260) (SARA 313) (CERCLA)
Ingredient Sequence Number: 08
Percent: <0.05
NIOSH (RTECS) Number: TQ1362000
CAS Number: 11096-82-5
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1262)
Ingredient Sequence Number: 09
Percent: <0.05
NIOSH (RTECS) Number: TQ1364000
CAS Number: 37324-23-5
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: POLYCHLORINATED BIPHENYL (AROCLOR 1268)
Ingredient Sequence Number: 10
Percent: <0.05
NIOSH (RTECS) Number: TQ1366000
CAS Number: 11100-14-4
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Proprietary: NO
Ingredient: SOLVENT; (INDUSTRIAL GRADE TRANSFORMER OIL)
Ingredient Sequence Number: 11
Percent: 99.9
NIOSH (RTECS) Number: 1000092SS
OSHA PEL: N/K (FP N)
ACGIH TLV: N/K (FP N)

Physical/Chemical Characteristics

Appearance And Odor: CLEAR YELLOWISH LIQUID; ODORLESS.
Melting Point: N/A
Specific Gravity: 0.889 (H*2O=1)
Solubility In Water: INSOLUBLE

Fire and Explosion Hazard Data

Flash Point: 300F,149C
Flash Point Method: COC
Extinguishing Media: DRY CHEMICAL, CO*2, WATER SPRAY OR REGULAR FOAM.
Special Fire Fighting Proc: USE NIOSH APPRVD SCBA & FULL PROT EQUIP (FP N). MOVE CNTNR FROM FIRE AREA IF YOU CAN DO IT W/OUT RISK. DO NOT SCATTER SPILLED MATL W/HIGH PRESS WATER (SUPDAT)
Unusual Fire And Expl Hazrds: MAY FORM CARBON MONOXIDE, PHOSGENE AND CARBONYL BROMIDE IN FIRE.

Reactivity Data

Stability: YES
Cond To Avoid (Stability): NONE.

Materials To Avoid: NONE SPECIFIED BY MANUFACTURER.
Hazardous Decomp Products: MAY FORM CARBON MONOXIDE, PHOSGENE AND CARBONYL BROMIDE IN FIRE.
Hazardous Poly Occur: NO
Conditions To Avoid (Poly): NOT RELEVANT

Health Hazard Data

[redacted]-LC50 Mixture: NONE SPECIFIED BY MANUFACTURER.
Route Of Entry - Inhalation: YES
Route Of Entry - Skin: YES
Route Of Entry - Ingestion: YES
Health Haz Acute And Chronic: SEVERAL COMPONENTS ARE ANIMAL POSITIVE, HUMAN SUSPECTED CARCINOGENS. PRIMARY IRRITANT. IRRITATES AND DAMAGES ALL TISSUES. MAY CAUSE LIVER, KIDNEY AND LUNG DAMAGE. MAY CAUSE CARDIAC ARRYTHIMIA, MAY SENSITIZE HEART TO EPINEPHRINE. MAY CAUSE ALLERGIC DERMATITIS OR CHLORACNE. MAY CAUSE CANCER OF LIVER, OR HEMATOPOETIC SYS.
Carcinogenicity - NTP: YES
Carcinogenicity - IARC: YES
Carcinogenicity - OSHA: NO
Explanation Carcinogenicity: AROCHLOR 1254:IARC MONOGRAPHS, SUPP, VOL 7, PG 322, 1987:GRP 2A. NTP 7TH ANNUAL RPT ON CARCINS, 1994:ANTIC TO BE(SUPDAT)
Signs/Symptoms Of Overexp: RED, DRY SCALY SKIN; CRACKING AND WEAPING SKIN; COUGH AND WHEEZING. JAUNDICE; NAUSEA AND VOMITING; UREMIA. MAY CAUSE CHLORACNE.
Med Cond Aggravated By Exp: DERMATITIS. LIVER DISEASE, KIDNEY DISEASE, ANEMIAS AND LEUKOPENIAS.
Emergency/First Aid Proc: INHALATION:REMOVE TO FRESH AIR. SUPPORT BREATHING (GIVE O*2/ARTF RESP) (FP N). EYES:IMMEDIATELY FLUSH W/RUNNING WATER FOR AT LEAST 15 MINUTES. SKIN:WASH W/SOAP & WATER. REMOVE AND ISOLATE CONTAMINATED CLOTHING AND SHOES AT THE SITE. INGESTION:GIVE SYRUP OF IPECAC 60CC WITH 180CC WATER IF SWALLOWED.

Precautions for Safe Handling and Use

[redacted]ns If Matl Released/Spill: DAM UP AND ABSORB. VENTILATE THE AREA. CALL [redacted]N UP TEAM. DO NOT WASH TO DRAIN.
[redacted]lizing Agent: NONE SPECIFIED BY MANUFACTURER.
[redacted]ste Disposal Method: DISPOSAL MUST BE I/A/W FEDERAL, STATE & LOCAL REGULATIONS (FP N). ABSORB AND INCINERATE OR DISPOSE AS HAZARDOUS WASTE.
Precautions-Handling/Storing: USE OR STORE ONLY IN AREAS WHERE SPILLS CAN BE CONTAINED.
Other Precautions: HANDLE WITH CARE! MATERIAL CONTAINS CARCINOGENS.

Control Measures

Respiratory Protection: NIOSH APPROVED ORGANIC VPAOR CARTRIDGE, FULL FACE PIECE OR SELF CONTAINED OR AIR SUPPLIED RESPIRATOR.
Ventilation: LOCAL EXHAUST &/OR MECHANICAL: USE IN HOOD. VENTILATE SPILL.
Protective Gloves: VITON OR NEOPRENE GLOVES.
Eye Protection: ANSI APPROVED CHEM WORKERS GOGGS (FP N).
Other Protective Equipment: EYE WASH FOUNTAIN & DELUGE SHOWER WHICH MEET ANSI DESIGN CRITERIA (FP N). CHEM IMPERVIOUS CLTHG IF LG AMTS IN (SUPDAT)
Work Hygienic Practices: USE CAREFUL LABORATORY TECHNIQUE. AVOID CONTACT.
Suppl. Safety & Health Data: FIRE FIGHT PROC:STREAMS. DIKE FIRE CONTROL NTP 7TH ANNUAL RPT ON CARCINS, 1994:ANTIC TO BE CARCIN. ANIMAL:LIVER. OTHER PROT EQUIP:USE. LAB COAT, IMPERVIOUS APRON W/SLEEVES AND CLOSED SHOES.

Transportation Data

Disposal Data

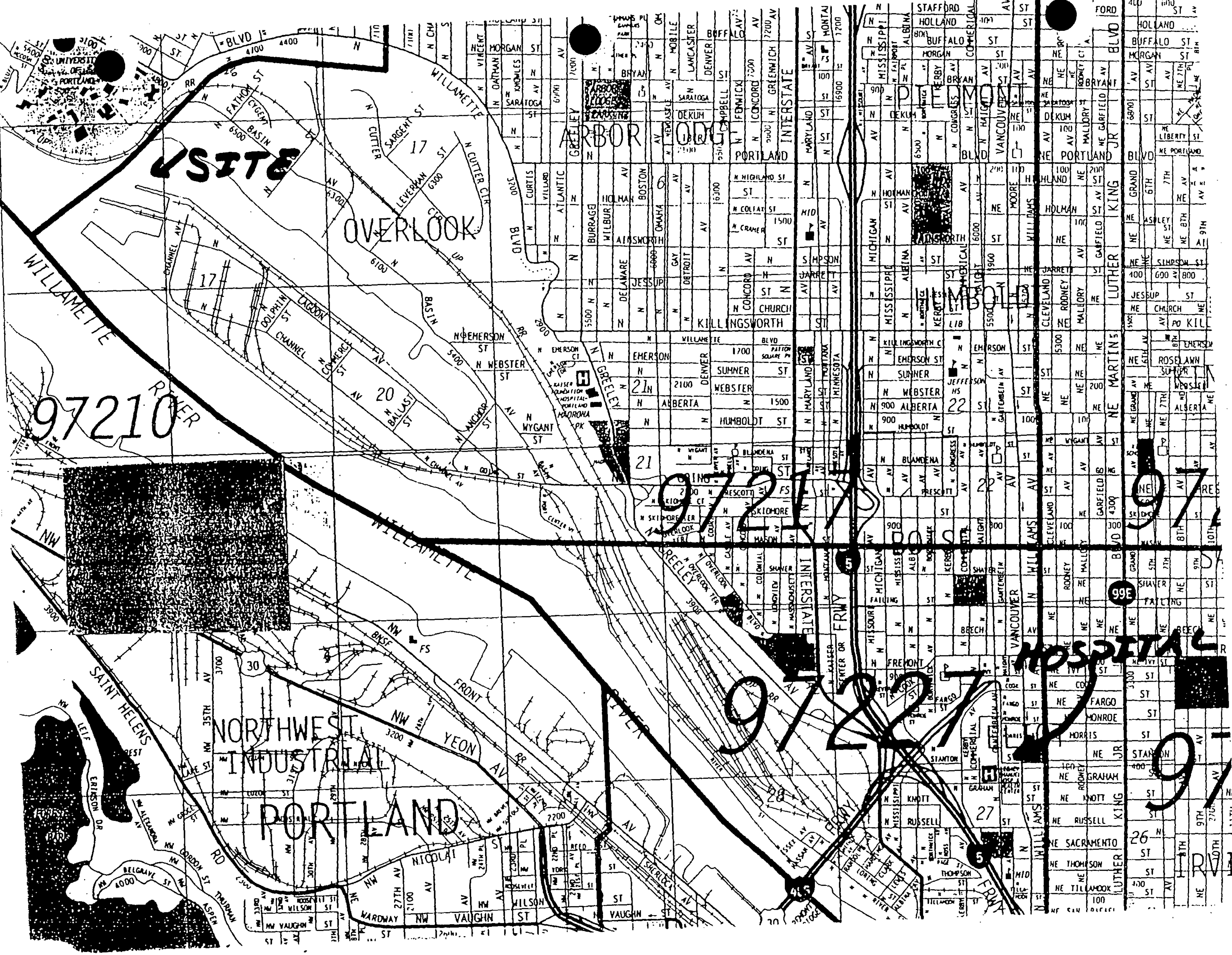
Label Data

[redacted] Required: YES
[redacted]al Review Date: 15AUG96
[redacted]ate: 07AUG96
Label Status: G
Common Name: PCB'S IN OIL, PT51
Chronic Hazard: YES

Signal Word: DANGER!
Acute Health Hazard-Severe: X
Contact Hazard-Severe: X
Fire Hazard-Slight: X
Reactivity Hazard-Slight: X
PRIMARY IRRITANT. IRRITATES AND DAMAGES ALL TISSUES. RED, DRY SCALY SKIN; CRACKING AND WEAPING SKIN; COUGH AND WHEEZING. JAUNDICE; NAUSEA AND VOMITING; UREMIA. MAY CAUSE CHLORACNE. CHRONIC:CANCER HAZARD. CONTAINS AROCHLORS, WHICH ARE LISTED AS AN ANIMAL LIVER CARCINOGEN (FP N). SEVERAL COMPONENTS ARE HUMAN SUSPECTED CARCINOGENS. MAY CAUSE LIVER, KIDNEY AND LUNG DAMAGE. MAY CAUSE CARDIAC ARRYTHIMIA, MAY SENSITIZE HEART TO EPINEPHRINE. MAY CAUSE ALLERGIC DERMATITIS OR CHLORACNE. MAY CAUSE CANCER OF LIVER, OR HEMATOPOETIC SYSTEM.
Protect Eye: Y
Protect Skin: Y
Protect Respiratory: Y
Label Name: ENVIRONMENTAL RESOURCE ASSOCIATES
Label Street: 5540 MARSHALL ST
Label City: ARVADA
Label State: CO
Label Zip Code: 80002
Label Country: US
Label Emergency Number: 303-431-8454

ATTACHMENT D-2

Hospital Map



SITE
HOSPITAL
97210
97217
97227
OVERLOOK
ARBOR LODGE
PIEDMONT
HUMBOLDT
NORTHWEST INDUSTRIAL
PORTLAND
WILLAMETTE RIVER
UNIVERSITY OF PORTLAND
INTERSTATE AV
GREELEY
KILLINGSWORTH ST
ALBERTA
VANCOUVER
WILLIAMS
MARTIN LUTHER KING JR BLVD
SAINT HELENS RD
NW YEON AV
NW FRONT AV
NICOLAI
NW VAUGHN ST

APPENDIX E – STRIPLIN ENVIRONMENTAL ASSOCIATES HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

PORTLAND SHIPYARD
REMEDIAL INVESTIGATION

DRAFT

December 14, 1999

Prepared by:

Striplin Environmental Associates, Inc.
222 Kenyon Street N.W.
Seattle, WA 98502

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1. INTRODUCTION

Striplin Environmental Associates, Inc. (SEA) is under contract with Bridgewater Group and the Port of Portland to complete the sediment portion of the Portland Shipyard (PSY) Remedial Investigation (RI). The field investigation will take place on the Willamette River and in Swan Island Lagoon adjacent to the Cascade General ship repair facility, Portland, Oregon (Figure 1). Cascade General currently operates the shipyard under contract with the Port of Portland. SEA's responsibilities in the field include collecting subsurface sediment cores and surface sediment grabs, processing sediment onboard the sampling vessel or onshore, and properly storing and transferring samples to participating laboratories.

This health and safety plan describes required and recommended procedures to help ensure the health and safety of SEA project personnel. A copy of this plan shall be maintained in a designated area on the sampling vessel and at the sample processing facility at all times. All personnel employed or contracted by SEA in support of sediment collection activities must read, understand, and comply with the information and procedures provided in this plan. Visitors must also read and comply with the plan. The plan must be read before a participant undertakes field activities; if any part is unclear, clarification should be sought from one of the safety officers before going to the field. Once the information has been read and understood, the individual must sign the Acknowledgment Form (Form 1, Appendix A), which will then be placed in the project file.

All subcontractors to SEA will prepare their own health and safety plan that is at least as protective as this plan, or they may adopt this plan as their own. If the subcontractor elects to prepare their own health and safety plan, copies of their plan and a signed acknowledgment form must be provided to the SEA project safety officer prior to the subcontractor's involvement in any field activities.

The following subjects are addressed by the health and safety plan: existing site conditions, description of sampling activities, hazard evaluation, hazard monitoring and control, emergency procedures, and worker training requirements. This plan may be modified at any time, based on the judgment of the site safety officer or the project safety officer. Any modification will be presented to the onsite team during a safety briefing and will be entered in Form 2 (Appendix A). Form 2 must be acknowledged by all sampling and processing personnel.

2. SITE CHARACTERISTICS

The Cascade General ship repair facility is located at the north end of Swan Island on the Willamette River (Figure 1). The property was the location of the original Portland airport, but since the 1940's the primary land use has been related to shipbuilding and repair. These activities may have resulted in the potential release of chemicals to the Willamette River and bottom sediments. The primary objective of the sediment characterization project is to determine the areal and vertical extent of any contaminated sediment in the vicinity of Cascade General ship repair facility.

The project area is shown in Figure 2 and includes the Swan Island Basin and Port of Portland turning basin. Previous sediment data collected in this vicinity include investigations conducted by SEA for the Port of Portland and Cascade General, Inc (SEA 1998), Weston for the Oregon Department of Environmental Quality and EPA Region 10 (DEQ 1997), and Dames and Moore for Cascade General, Inc (Dames and Moore 1998). Based on preliminary review of existing information, chemical data most relevant to health and safety concerns and potential chemical exposure are summarized below.

Sediment types in the project area are expected to range from fine-grained material (i.e. silts and clays) in the Swan Island Basin to very coarse (sandblast grit) at the dry docks. Chemicals indicating potential concerns for the health and safety of field personnel were identified based on frequency of detection, enrichment ratios, and comparison to AETs in surface sediments (SEA 1998). These include the following:

Arsenic
Cadmium
Copper
Lead
Mercury
Nickel
Zinc
High-molecular weight polycyclic aromatic hydrocarbons (HPAH)
Low- molecular weight polycyclic aromatic hydrocarbons (LPAH)
Polychlorinated biphenyls (PCBs)
Tributyl tin

Protective measures to address the hazards associates with these listed chemicals are expected to provide sufficient protection for any other chemicals that may be present in the sediments.

3. DESCRIPTION OF SEDIMENT SAMPLING ACTIVITIES

Two types of sediment samples will be collected: subsurface cores and surface grabs. Sediment cores will be collected to document the physical and chemical characteristics of subsurface sediments in the project area. Sediments will be collected using Marine Sampling Systems (MSS) vibracorer operated from the vessel *R/V Nancy Anne*. Cores will be acquired by SEA personnel or their contractors using SEA guidelines and vessel-specific requirements. The cores will be transported to an onshore sampling facility where they will be opened and sampled by SEA personnel or contractors. All sediment sampling methods will follow standard regional guidelines (PSEP 1986, 1989a, 1989b, 1995; PSDDA 1988, 1989) and the project sampling and analysis plan (SAP). Core sediment will be described in a core log form and sampled according to the scheme described in the project SAP. Sediments will be placed into decontaminated stainless steel bowls and homogenized until the sediment is uniform in texture and color. The sediment is then placed into jars and shipped to the chemical testing laboratory.

Surface sediments will be sampled using a double van Veen grab or hydraulically-powered van Veen grab. The grab will be retrieved and deployed by SEA personnel with assistance from the *R/V Nancy Anne* vessel crew. Sediment will be collected from the grab and processed on board the vessel. Sediments will be placed in decontaminated stainless steel bowls, homogenized, placed in jars and shipped to the chemical testing laboratories.

Surface or subsurface sediment sampling in areas that cannot be accessed by the sampling vessel (e.g. under docks) may be sampled by divers using hand-held samplers or coring devices. Diving services will be provided by subcontractor(s) to SEA. The diving subcontractor will be fully responsible for all health and safety procedures related to diving operations. Therefore, specific requirements for diving safety are not addressed in this health and safety plan.

4. HAZARD EVALUATION

The overall hazard level associated with the planned sediment sampling activities is low. Hazards encountered during this sampling program are due to physical safety hazards associated with the field operations and potential exposure to hazardous materials present within the sediments. As described below, protective equipment and safe working procedures will help prevent accidents caused by these hazards. Exposure to harmful microbial organisms or other organisms in the sediments is not expected during this program; therefore, biological hazards are not discussed.

4.1. CHEMICAL HAZARDS

4.1.1. Potential Chemical Hazards in Sediments

Chemicals identified at elevated concentrations in the project area include numerous metals, PAHs, PCBs and TBT. These chemicals are relatively nonvolatile and pose a low risk for inhalation. Chemicals will be bound in a wet solid matrix, and personnel will be working in the open or in well-ventilated areas. Nonetheless, many of these compounds are considered carcinogenic and exposure by all routes (including dermal and ingestion) should be minimized. Material safety data sheets or occupational health guidelines for these compounds are provided in Appendix B.

Respiratory and inhalation hazards commonly occur during under dusty (dry) and windy conditions - conditions not likely to be found while handling sediment samples as part of this sediment characterization. No volatile organic compounds were detected during the initial phase of the sediment characterization (SEA 1998). In addition, since all work will be conducted in the open air or well-ventilated areas, respirator use is not anticipated and air monitoring will not be required. The sample-processing leader at the onshore processing facility should ensure that fans, doors, and windows allow adequate ventilation. Core processing will be moved to a covered outdoor area if the facility does not allow adequate fresh air circulation.

Sediments containing metals, PAHs, and other organic compounds in contact with the skin and eyes can cause irritation and burning. As a precautionary measure, gloves will be worn at all times when sampling and processing sediments. To avoid accidental ingestion of chemicals present in the sediment or while decontaminating sampling gear, gloves in contact with potential hazardous chemicals should not come in contact with the facial area. Additional protective measures are described in Section 5.

Hydrogen sulfide, a naturally occurring gas emitted from marine sediments, is potentially toxic via inhalation, ingestion, and skin and eye contact. Inhalation can result in respiratory

irritation, rhinitis, and edema of the lungs. Subacute exposures to hydrogen sulfide may result in headache, dizziness, staggering gate, and agitation. As sampling personnel will be processing sediments in open air or well-ventilated areas, hydrogen sulfide toxicity is not expected.

4.1.2. Chemicals Used During Sample Collection

The following chemicals will be used as sample preservatives or for decontamination of sampling gear and implements. Detailed information is provided in Appendix B.

Zinc acetate. A 2 Normal (2N) solution of zinc acetate is used to preserve sediment samples for sulfide analysis. Precautions will be taken when using zinc acetate. Zinc acetate will be pre-measured into sample jars under proper ventilation prior to sediment collections. Jars containing zinc acetate will be sealed and transported in plastic bags.

Nitric acid (0.1 Normal): Nitric acid will be used to strip trace amounts of metals from sampling equipment to prevent cross contamination. It is a clear, colorless liquid with a strong odor. Nitric acid will burn exposed skin on contact. Personnel are required to wear protective gloves and goggles whenever handling this decontaminating agent. The dilute nitric acid solution is used in open, well-ventilated areas only. Respirators will be available but are optional, and may be worn if desired by SEA personnel.

Methanol: Methanol is a volatile solvent that will be used to strip organic compounds from sampling equipment. It is a clear, colorless liquid with a strong odor and is highly flammable. Health effects are similar to those described above for volatile organic compounds. Ingestion of methanol can result in blindness. Personnel are required to wear protective gloves and goggles whenever handling this decontaminating agent. It is to be used in the open-air only. Respirators will be available but are optional, and may be worn if desired by field personnel.

4.2. PHYSICAL HAZARDS

4.2.1. Research Vessel Operations

The physical hazards associated with the deployment and retrieval of large pieces of sampling equipment result from their weight and the method of deployment. Only persons whose presence is required will be allowed on the deck during deployment and retrieval of the samplers. Under circumstances of potentially dangerous waves or winds, the vessel pilot and cruise leader will employ best professional judgment to ensure safe field operations. Emergency procedures for a man-overboard are discussed in Section 6.3.

To avoid injuries from deck gear and equipment, all personnel on the deck will wear hard hats and steel-toed boots. Personnel will also wear Coast Guard-approved life vests when sampling during adverse weather conditions, handling equipment over the rail, or if directed by the vessel operator or cruise leader. Sample handling equipment, containers, deck lines, and water hoses

not in immediate use will be kept clear of walkways and work areas until needed. any sediment on the deck after operations at a given station are completed will be washed overboard to prevent slipping.

4.2.2. Physical Exposure

Exposure to the elements and fatigue are two major causes of accidents onboard boats. The sampling events may include long work days, and the weather can be unpredictable. Working in cold, rough waters can lead to sea sickness, exposure and fatigue. The combination of rough waters and fatigue increases the chances for a man-overboard situation. To prevent fatigue and overexposure in adverse weather conditions, field personnel will take regular work breaks. Extra clothing will be brought to accommodate changes in weather. Cold stress can be manifested as hypothermia (discussed further in Section 6.4). Heat-related illnesses can occur at any time when protective clothing is worn. When temperatures average 70-75°F, the risk of heat-related illnesses increases. Heat stress can be manifested as both heat stroke and heat exhaustion (discussed further in Section 6.5). Life vests are available for all personnel working on the deck and must be donned when directed by the safety officer or vessel operator. The vessel is also equipped with life rings, and each crewmember will be briefed on their storage location. Fatigue also presents a hazard when working at sea. It can be compounded by the motion of the vessel, exposure, or heat stress. Personnel should monitor their own condition and capabilities and should be responsible for taking appropriate measures (discussed below) to relieve fatigue, exposure, or heat stress. The chief scientist/safety officer and vessel operator can also direct any member of the crew to cease working.

5. HAZARD MONITORING AND CONTROL

5.1. PERSONNEL AND ORGANIZATION

For this sediment sampling effort, project organization and health and safety responsibilities for SEA employees for this sampling program will be as follows.

Project Safety Officer/Cruise Leader. Pete Striplin, SEA, will serve as project safety officer. He will be in charge of all sampling and sample processing activities, and is responsible for health and safety procedures, including:

- Ensuring that all workers know and follow the project health and safety plan
- Conducting health and safety meetings or briefings, as necessary
- Evaluating and modifying the level of protective apparel and equipment, as necessary, based on site conditions.
- Verifying compliance with the health and safety plan and applicable health and safety regulations
- Completing a Project Health and Safety Evaluation form (Appendix A, Form 4) upon completion of the field effort and returning this form to the SEA company safety officer.

Site Safety Officers. Tom Schulz and Dave Browning, SEA, will serve as safety officers onboard the sampling vessel and at the onshore sample processing facility. Their health and safety responsibilities are as follows:

- Ensuring that all workers know and follow the project health and safety plan
- Establish work zones onboard the vessel and at the sample processing facility and ensure that all workers are informed as to their location. Form 2, Modification to Health and Safety Plan, must be completed for work zones at the sample processing facility.
- Evaluating and modifying the level of protective apparel and equipment, as necessary, based on site conditions.
- Verifying compliance with the health and safety plan and applicable health and safety regulations

Field Staff. All SEA employees and contractors will be responsible for knowing and implementing the policies and procedures stated in the project health and safety plan, including:

- Complying with proper safety and health practices, as stated in the plan and training
- Using required safety devices and personal protective equipment
- Notifying a supervisor of unsafe conditions or acts immediately
- Reporting all accidents to a supervisor promptly, regardless of the severity of injury
- Carrying out all work in a manner not endangering to any employee

Company Health and Safety Officer. Vicki Fagerness, SEA, is responsible for administering the company health and safety program. She will:

- Prepare the site-specific project health and safety plan and ensure that it is consistent with the company health and safety program
- Work with field safety officers to implement appropriate health and safety practices and eliminate hazards.

President. Betsy Striplin, SEA's company president, has final authority over all workplace and hazardous waste site operations. She will:

- Ensure that adequate resources are available to implement the health and safety plan
- Ensure that employees are implementing their responsibilities as defined in the plan
- If necessary, ensure that accidents are fully investigated and corrective action taken to prevent recurrence of the hazardous conditions or behaviors.

5.2. PERSONAL PROTECTIVE CLOTHING AND EQUIPMENT

On board the sampling vessel, protective gear includes regular work clothes with coated rain pants. Splash protection may be upgraded to coated rain jacket and pants, taped at the wrists and ankles if appropriate, as determined necessary by the cruise leader. Also included are the following:

- Nitrile outer gloves
- Polypropylene, nitrile, or polyethylene inner gloves
- Steel-toed, chemically resistant, impermeable outer boots
- Hard hat (when overhead hazards are present)
- Safety glasses or goggles when conditions for splash exposure exist
- Hearing protection will be available upon request

Each crewmember is expected to bring clothing appropriate to the weather and work task to minimize the hazards of exposure and heat stress. Flotation vests will be provided and may be worn by SEA personnel at their own discretion unless otherwise required by the cruise leader or vessel operator, weather or work conditions. Personnel are not required to use respirators unless the cruise leader recommends otherwise.

At the sample processing facility, protective clothing includes regular work clothes with coated

rain pants, coveralls, or lab coats. Impermeable boots are recommended; however, at minimum, footwear must be of a material that will allow decontamination and removal of all sediment by washing. Nitrile outer gloves and inner gloves will be worn when handling cores and sediment.

5.3. MONITORING AND SAMPLING PLAN

No air monitoring equipment is necessary for sediment sampling on the Willamette River (see Section 4.1.1). All sediment sampling will take place in the open air (i.e. on the vessel deck) or in a well-ventilated area.

5.4. SITE CONTROL MEASURES

5.4.1. On-board the Vessel

The deck of the vessel is designated as the work zone. Personnel are expected to use direct line-of-sight and open communications while in the work zone.

The exclusion zone is the area where contamination could or does occur. During coring or grab sample collection, the exclusion zone will be the area of the vessel in which sediments are being handled. When no sediment is onboard, the entire vessel is the Support Zone. Only authorized field personnel will be allowed in the exclusion zone.

The contamination reduction zone (CRA) is the transition area between the contaminated area and the clean area. The CRZ during coring and grab sample collection is the vessel deck, except as noted in the preceding paragraph. Decontamination of both personnel and equipment will occur in this zone.

The support zone is where all personnel will suit-up in personal protective equipment before entering the exclusion zone. The support zone will be onshore, in the cabin, or on the vessel deck when contaminated sediment is not on deck.

5.4.2. Onshore Processing Facility

Exclusion, contaminant reduction, and support zones in the sample processing area will be established based on the facility layout, space requirements and access to wash water. Work zones will be established by the site safety officer prior to beginning work and described using Form 2 (Appendix A) for Modification to the Health and Safety Plan. The sample processing task leader and the site safety officer will ensure that all field personnel are informed of work zone locations at the processing facility.

5.4.3. Other Safety Procedures

All personnel working in the field will follow the rules and procedures listed below:

- Before any field sampling begins, all personnel must review the project health and safety plan, become familiar with the required safety procedures, and sign the plan acknowledgment form.

- Copies of the health and safety plan will be available on board the vessel and at the onshore sample processing area.

- The vessel operator and cruise leader will monitor weather condition and forecasts. Either the vessel operator or the cruise leader has the authority to halt operations if conditions are deemed to be unsafe.

- No eating, drinking or smoking will be allowed in the exclusion or contamination reduction zones

- The buddy system will be used during all sampling activities

5.5. DECONTAMINATION

All sampling equipment will be decontaminated as described in the project SAP:
- Rinse with tap water or water provided by the sampling vessel
- Wash with brush and Alconox detergent
- Double rinse with distilled water
- Rinse with 0.1 N nitric acid
- Rinse with distilled water
- Rinse with methanol (omit this step if the equipment will be used to sample VOAs).

Impermeable personal protective gear will be decontaminated as follows:
- Rinse boots, raingear and outer gloves with water
- Scrub with brush and Alconox detergent
- Rinse with tap water or water provided by the sampling vessel

Impermeable protective gear will be decontaminated before removal. Outer gloves will be removed first, followed by raingear and boots, then inner glove liners. Hands should then be thoroughly washed.

At the onshore core processing facility, the same decontamination procedures will be followed. If coveralls or lab coats are worn instead of impermeable rain gear, clothing will be removed before leaving the sample processing facility, placed in a plastic bag, and laundered as soon as possible.

5.6. INVESTIGATION DERIVED WASTE MANAGEMENT PLAN

All sediments or other site-derived materials (e.g., wood debris) will be discarded overboard at the work site. Wash water on the vessel will also be discarded overboard. Wash water at the onshore core processing facility will be disposed to the city sanitary sewer system. No other waste disposal is anticipated.

5.7. VISITORS

Authorized visitors will only be allowed to observe operations from the support zone and must obey all instructions of the cruise leader. Exceptions to this are representatives from government agencies or other members of the project consultant team who establish that they possess training consistent with the requirements of this plan. They must also possess appropriate health and safety equipment at the time of the visit, have a health and safety plan at least as stringent as this plan, or adopt this plan as their own.

6. EMERGENCY PLAN

The project safety officer will confirm key emergency services prior to the start of sampling activities. The purpose of this information exchange is twofold: to establish final procedures for use in emergencies and to inform outside help of the activities being performed onsite and the associated potential problems. The U.S. Coast Guard and/or marine traffic controllers will be informed, as necessary, of the operations and locations of the research vessel.

6.1. REPORTING/NOTIFICATION PROCEDURES

In the case of any emergency, the SEA project safety officer is to be notified immediately. The safety officer will initiate contacts as follows:

1) Call appropriate emergency services numbers (ambulance, fire, etc.) if not already done. Provide the following information:
    - Name and location of person reporting
    - Location of accident/incident
    - Name and affiliation of injured party
    - Description of injuries
    - Status of medical aid effort
    - Details on any chemicals involved and description of any personnel or contaminated gear to be sent with the injured party
    - Summary of the accident, including the suspected cause and the time of occurrence
    - Temporary control measures taken to minimize further risk.

    Note: This information is not to be released under any circumstances to parties other than the cruise leader and bona fide emergency response team members.

2) Call SEA Company Safety Officer and provide information noted in Step 1 above.

3) The project safety officer or other delegated person will complete a written accident/incident report using Form 3 (Appendix A) within 24 hours. This report should be submitted to the SEA Safety Officer.

The following resources are to be used in cases of emergency:

- **Emergency Contacts:** Table 1 includes both the appropriate emergency services (top of table) and the appropriate project contacts (bottom of table).
- **Nearest Phone:** A cellular telephone will be on the vessel. A phone will also be available at the onshore processing facility.
- **Onsite Emergency Equipment:** A first aid kit, portable fire extinguisher, and an eyewash kit will be located on the vessel and at the onshore processing facility.
- **Offsite Emergency Services:** Phone numbers for offsite emergency services are listed in Table 1. Copies of this table will be located on the vessel and at the onshore processing facility.

6.2. HOSPITAL ROUTES

Kaiser-Permanente - Bess Kaiser Medical Center

5055 N. Greeley
Portland, Oregon 97217

General: (503) 285-9321
Emergency (all hours): (503) 285-9321

If an injury requiring immediate medical assistance occurs on the vessel, the injured person should be transported to the hospital by ambulance. If medical care is required, but an ambulance service is not warranted, field vehicles may be used to transport the injured person to the hospital. It is anticipated that the sampling vehicles will be parked at Port of Portland facilities at Swan Island. It is also likely that the onshore sample processing facility will be located on Swan Island at one of the Port of Portland buildings.

If ambulance service is required:

If the accident occurs on the water, the sampling vessel should proceed directly to the Port of Portland dock or nearest accessible location. Emergency services should be immediately contacted using the cell phone to arrange pickup at the dock or the onshore processing facility.

Directions from the dock or onshore sample processing facility to the hospital (Figure 3):

From the Port of Portland dock or the sample processing facility, proceed south on N. Lagoon Avenue. Turn right (west) onto N. Anchor St., and take the next (slight) left onto N. Channel Ave. Travel approximately 0.5 miles, noting that N. Channel Ave. becomes N. Going St. Exit right onto the ramp and then right again onto Greeley Blvd. Travel north approximately 0.3 miles. The hospital and emergency room entrance are on the left.

6.3. MAN OVERBOARD

While the team is working over water on the research vessel, or using heavy equipment (e.g., subsurface coring), or during stormy weather, there is a potential for a man-overboard situation. If this situation occurs, all vessel engines will be stopped immediately. Flotation devices (e.g.,

life rings) attached to lines will be thrown to the victim from the vessel. The victim will then be brought aboard the research vessel or towed to shore; wet clothes will be removed and replaced with dry clothing. The victim may need to be treated for cold stress (discussed below). No other person shall enter the water unless the victim is unconscious or seriously injured. Rescuers must wear life preservers and be tethered to the research vessel or shore.

6.4. COLD STRESS

In cold weather conditions, field teams must be prepared to wear proper protective clothing and to recognize symptoms of cold stress. Cold stress can be manifested as both hypothermia and frostbite.

- **Hypothermia**, a cold-induced decrease in the core body temperature, can decrease attentiveness and manual dexterity. Hypothermia produces shivering, numbness, drowsiness, muscular weakness, and, if severe enough, death.

  Treatment: A victim of hypothermia should be taken indoors (or vessel's cabin) quickly. Provide rapid but gentle warming. Remove wet or cold garments and provide warm, dry clothing or covering. Dry the person thoroughly. If the victim reacts and is conscious, give a hot drink. It may be necessary to wrap the victim together with warm water bottles, or persons in blankets, or a sleeping bag. Seek immediate medical care.

- **Frostbite** results from the constriction of blood vessels in the extremities, and decreases the supply of warming blood to these areas. This drop in blood supply may result in the formation of ice crystals in the tissues, causing tissue damage. The symptoms of frostbite are white or grayish skin, blisters, or numbness.

  Treatment: Bring the victim indoors and rewarm the areas quickly in water of 102°F to 105°F. Give a warm drink – not coffee, tea, or alcohol. The victim should not smoke. Smoking tends to constrict the blood vessels in the skin, making the injury slow to heal. Keep the frozen parts in warm water or covered with warm cloths for 30 minutes. Then elevate the injured area and protect it from injury. Do not allow the blisters to be broken. Use sterile, soft, dry material to cover the injured areas. Keep the victim warm and get immediate medical care.

6.5. HEAT STRESS

Heat stress is not anticipated during winter or spring sampling, but heat-related illnesses can occur at any time when protective clothing is worn. If site activities take place when temperatures average 70-75°F, the risk of heat-related illnesses increases. Heat stress can be manifested as both heat stroke and heat exhaustion:

- In **heat stroke**, the person's temperature control system that causes sweating stops working correctly. The body temperature rises so high that brain damage and death will result if the person is not cooled quickly. The main signs of heat stroke are red or flushed skin; hot, dry skin, although the person may have been sweating earlier; and extremely high body temperature, often to 106°F (41°C). There may be dizziness, nausea, headache, rapid pulse, and unconsciousness.

Treatment: Cool a victim of heat stroke quickly. If the body temperature is not brought down fast, permanent brain damage or death will result. Soak the person in cool but not cold water, sponge the body with rubbing alcohol or cool water, or pour water on the body to reduce temperature to a safe level - about 102°F (39°C). Then stop cooling and observe the victim for 10 minutes. If the temperature starts to rise again, cool the victim again. Do not give coffee, tea, or alcoholic beverages. When the victim's temperature remains at a safe level, put the victim to bed and get medical help.

- **Heat exhaustion** is much less dangerous than heat stroke. The major signs of heat exhaustion are pale, clammy skin, profuse perspiration, and extreme tiredness or weakness. The body temperature is approximately normal. The person may have a headache and may vomit.

  Treatment: For mild heat exhaustion, provide bed rest. Give a salt solution (1/2 teaspoon salt in 1/2 glass of water) every 15 minutes for three or four doses. Medical care is needed for severe heat exhaustion.

7. TRAINING AND MEDICAL SURVEILLANCE

All personnel involved in the conduct of this program have completed the 40 hour hazardous waste site training and, if applicable, a current 8 hour refresher. (Current training certificates for all field staff are contained in Appendix C). All SEA personnel that will be sampling and handling sediments are participants in SEA's company medical monitoring program.

8. PLAN ACCEPTANCE

This site health and safety plan has been written for the use of SEA personnel. SEA claims no responsibility for its use by others. The plan is written for the specific site conditions, purposes, dates, and personnel specified and must be amended if these conditions change.

PLAN PREPARED BY: Striplin Environmental Associates

DATE: December 1999

ACCEPTED BY: _____ DATE: _____

ACCEPTED BY: _____ DATE: _____

ACCEPTED BY: _____ DATE: _____

9. REFERENCES

Puget Sound Dredged Disposal Analysis (PSDDA). 1988. Evaluation Procedures Technical Appendix - Phase I (Central Puget Sound). Puget Sound Dredged Disposal Analysis Reports Series. Cooperatively published by (in alphabetical order) U.S. Army Corps of Engineers, Seattle District; U.S. EPA, Region 10; Washington State Department of Ecology; and Washington State Department of Natural Resources.

Puget Sound Dredged Disposal Analysis (PSDDA). 1989. Management plan report - unconfined open-water disposal of dredged material, Phase II (North and South Puget Sound). Puget Sound Dredged Disposal Analysis Reports Series. Cooperatively published by (in alphabetical order) U.S. Army Corps of Engineers, Seattle District; U.S. EPA, Region 10; Washington State Department of Ecology; and Washington State Department of Natural Resources.

Puget Sound Estuary Program (PSEP). 1986. Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound. Final Report. TC-3991-04. Prepared for EPA, Region 10, Seattle, Washington. Tetra Tech, Inc., Bellevue, WA.

Puget Sound Estuary Program. 1989a. Recommended Protocols for Measuring Metals in Puget Sound Water, Sediment, and Tissue Samples. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

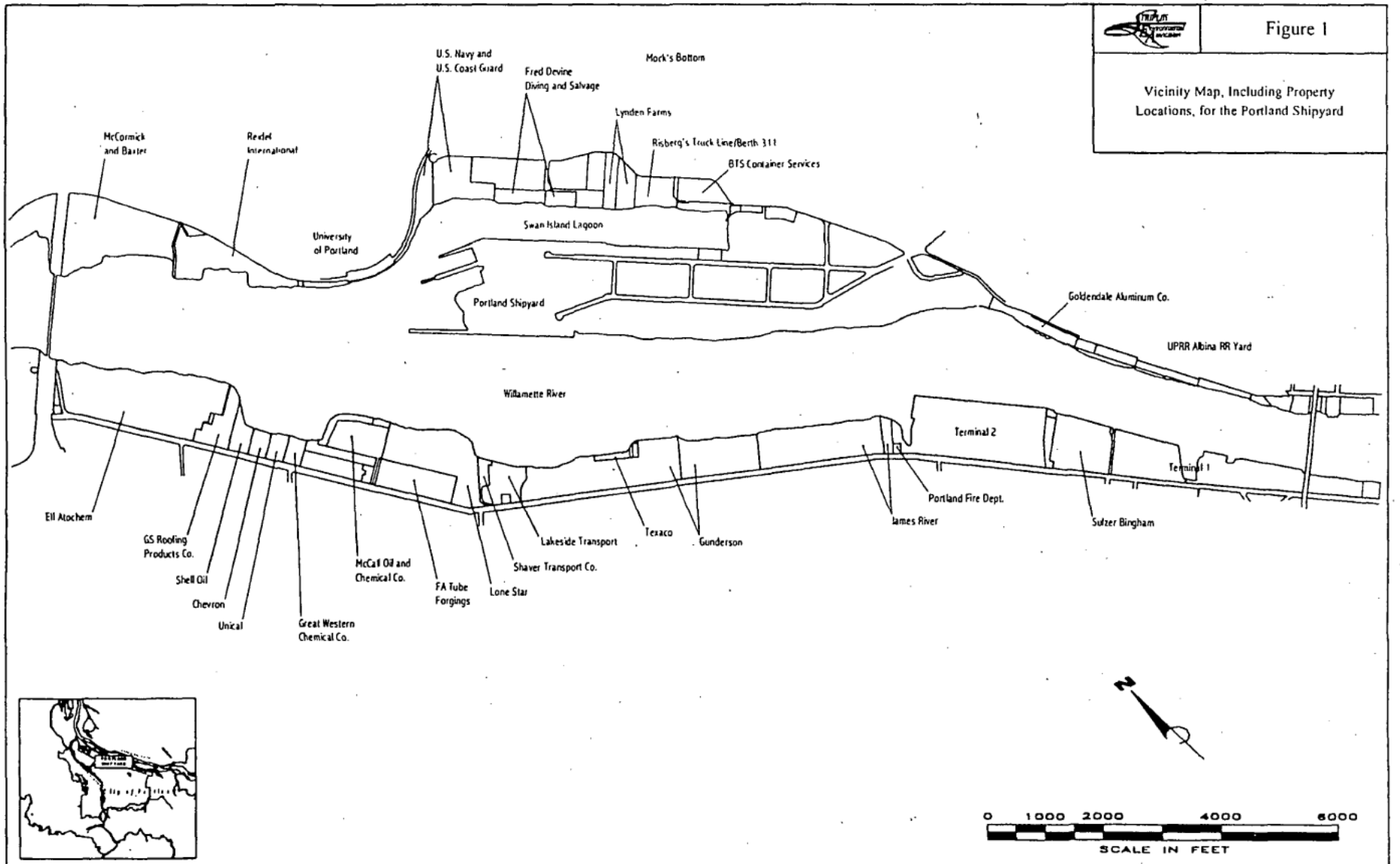
Puget Sound Estuary Program. 1989b. Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

Puget Sound Estuary Program (PSEP). 1995. Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments. Final Report. Prepared for EPA, Region 10, Office of Puget Sound, Seattle, Washington. Washington Department of Ecology, Olympia, WA.

Striplin Environmental Associates (SEA). 1998. Portland Shipyard Sediment Investigation Data Report. Prepared for Port of Portland and Cascade General, Inc., Portland, Oregon. Olympia, WA.

Figure 1

Vicinity Map, Including Property Locations, for the Portland Shipyard



Portland Shipyard Workplan	June 1998	PSY-WP Figure 01.ai	*PRIVILEGED AND CONFIDENTIAL: Work Product Prepared Under Attorney/Client Privilege*

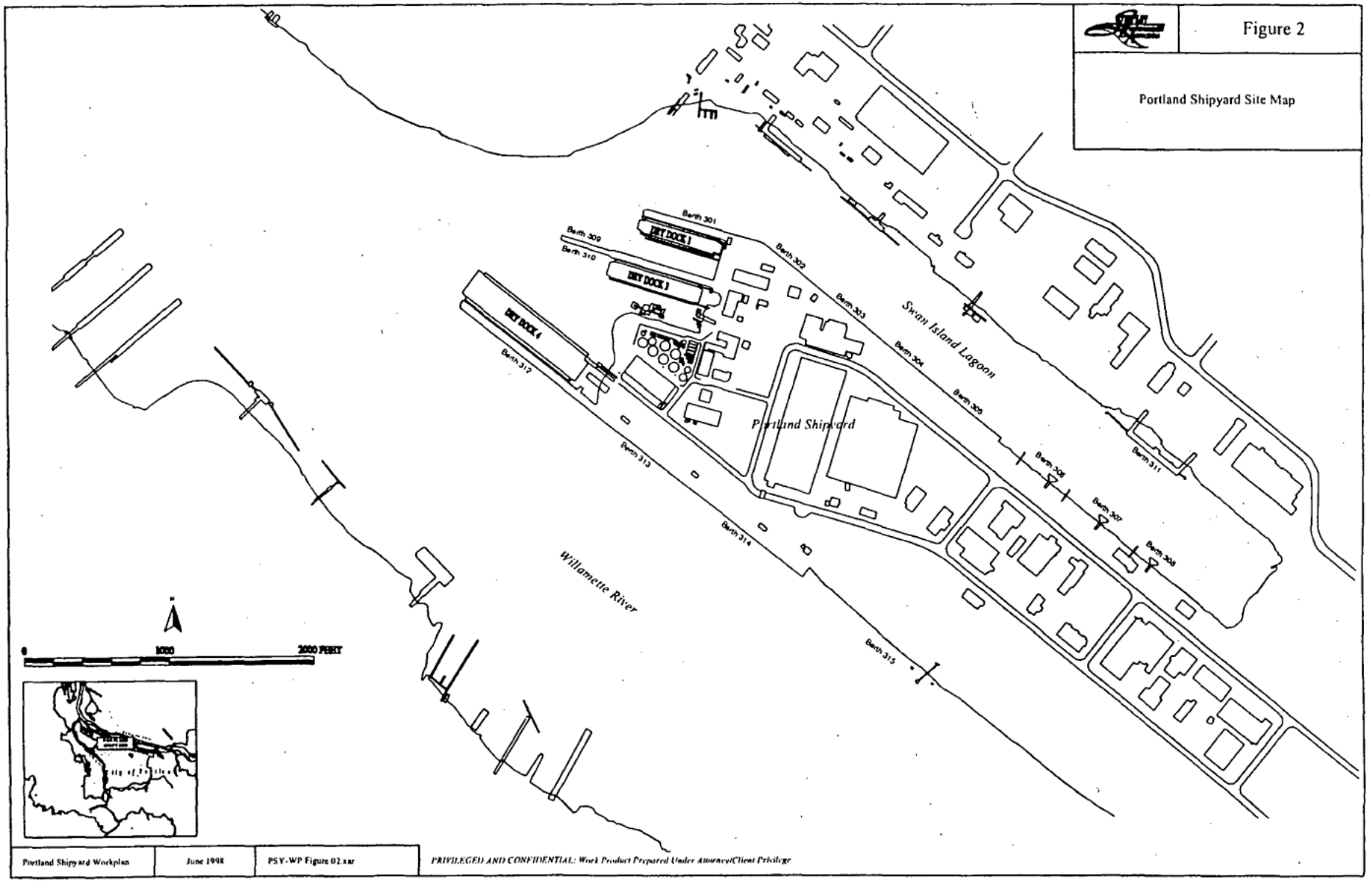


Figure 2
Portland Shipyard Site Map
Swan Island Lagoon
Portland Shipyard
Willamette River
Berth 301
Berth 309
Berth 310
Berth 302
Berth 303
Berth 304
Berth 305
Berth 306
Berth 307
Berth 308
Berth 311
Berth 312
Berth 313
Berth 314
Berth 315
Berth 317
DRY DOCK 1
DRY DOCK 2
DRY DOCK 4
0
1000
2000 FEET
Portland Shipyard Workplan
June 1998
PSY-WP Figure 02.xar
PRIVILEGED AND CONFIDENTIAL: Work Product Prepared Under Attorney/Client Privilege

DRAFT

5055 N Greeley Ave, Portland, OR 97217-3524

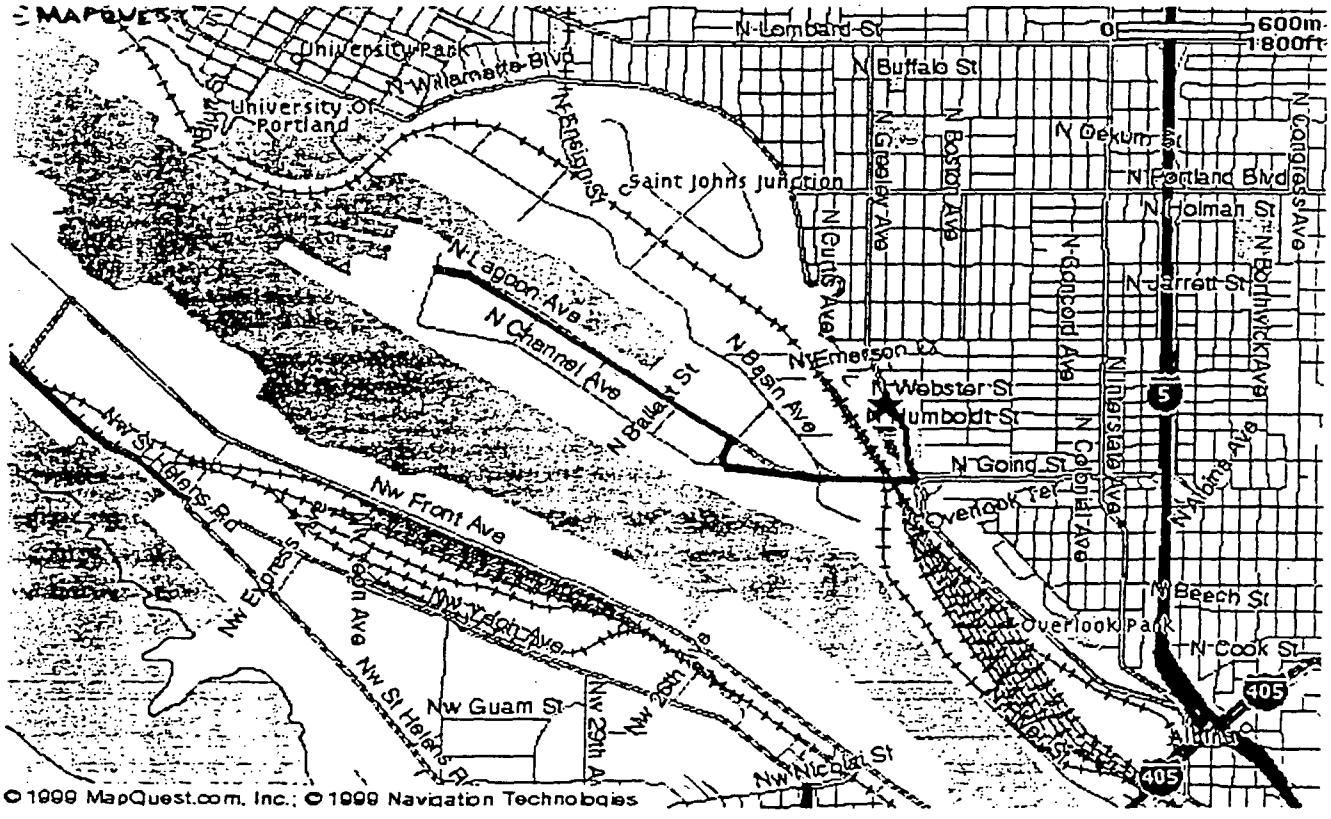


Figure 3. Transportation route from dock and onshore processing facility to hospital (source: www.MapQuest.com).

Table E-1. Emergency Services and Contacts.

Emergency Services

Service	Name/Location	Phone Number
Ambulance	---/---	911
Fire	---/---	911
Police	---/---	911
Hospitals	**Bess Kaiser Medical Center**	
	5055 N. Greeley	General: (503) 285-9321
	Portland, Oregon 97217	Emergency (503) 285-9321

Contact Information

SEA/Project Safety Officer	Home Phone	Work Phone	Location
Vicki Fagerness	(b) (6)	(360) 705-3983	SEA, Olympia, WA
Betsy Striplin		(360) 866-8343	SEA, Olympia, WA
Cruise Leader/Site Safety Officer			
Pete Striplin		(360) 866-8343	SEA, Olympia, WA

ATTACHMENT E-1

FORMS

FORM 1

ACKNOWLEDGMENT

I have read the attached Health and Safety Plan for the Port of Portland sediment characterization project. I have discussed any questions that I have regarding these materials with the SEA Safety Officer or Cruise Leader, and I understand the requirements of the Health and Safety Plan.

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

Employee _____ Date _____

FORM 2

MODIFICATION TO HEALTH AND SAFETY PLAN

DATE ___/___/___

Modification: _____

_____

_____

Reasons for Modification: _____

_____

_____

Site Personnel Briefed:

Name: _____ Date _____

Name: _____ Date: _____

Name: _____ Date: _____

Name: _____ Date: _____

Name: _____ Date: _____

Name: _____ Date: _____

Name: _____ Date: _____

Name: _____ Date: _____

Approvals

Site Safety Officer: _____

Project Safety Officer: _____

Project Manager: _____

Others: _____

FORM 3

EMPLOYEE EXPOSURE/INJURY INCIDENT REPORT

(Use additional page if necessary)

Date: _____ Time: _____

Name: _____ Employer: _____

Site Name and Location: _____

Site Weather (clear, rain, snow, etc.): _____

Nature of Illness/Injury: _____

Symptoms: _____

Action Taken: Rest _____ First Aid _____ Medical _____

Transported by: _____

Witnessed by: _____

Hospital's Name: _____

Treatment: _____

Comments: _____

_____

_____

What was the person doing at the time of the accident/incident? _____

_____

Personal Protective Equipment Worn: _____

_____

Cause of Accident/Incident: _____

_____

What immediate action was taken to prevent recurrence?_____

_____

Additional comments:

Employees Signature: _____ Date: _____

Supervisors Signature: _____ Date: _____

Site Safety Officers Signature: _____ Date: _____

ATTACHMENT E-2

MATERIAL SAFETY DATA SHEETS/OCCUPATIONAL HEALTH INFORMATION

Common Name: Arsenic
CAS Number: 7440-38-2
DOT Number: UN 1558
Date: November, 1986

HAZARD SUMMARY

* Arsenic can affect you when breathed in and may enter through the skin.
* Arsenic is a CARCINOGEN//HANDLE WITH EXTREME CAUTION.
* It may damage the developing fetus.
* Skin contact can cause burning, itching, thickening and color changes.
* High or repeated exposure can damage nerves, with "pins and needles," numbness, and weakness of arms and legs as well as poor appetite, nausea, stomach cramps, nose ulcers, hoarseness, or damage to the liver, blood vessels, or red blood cells.
* Arsenic near acid or acid mist can release a VERY DEADLY gas, Arsine.

IDENTIFICATION

Arsenic is a silver-gray brittle, crystalline solid. It also exists in black and yellow amorphous forms. It is used as an alloying agent for heavy metals, in special solders and in medicine.

REASON FOR CITATION

* Arsenic is on the Hazardous Substance List because it is regulated by OSHA and cited by ACGIH, NIOSH, IARC, DOT and other authorities.
* This chemical is on the Special Health Hazard Substance List because it is a CARCINOGEN.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work-related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 0.01 mg/m3 averaged over an 8-hour workshift.

NIOSH: The recommended airborne exposure limit is 0.002 mg/m3, not to be exceeded during any 15 minute work period.

ACGIH: The recommended airborne exposure limit is 0.2 mg/m3 averaged over an 8-hour workshift.

* The above exposure limits are for air levels only. When skin contact also occurs, you may be overexposed, even though air levels are less than the limits listed above.
* Arsenic is a CARCINOGEN in humans. There may be no safe level of exposure to a carcinogen, so all contact should be reduced to the lowest possible level.

WAYS OF REDUCING EXPOSURE

* A regulated, marked area should be established where Arsenic is handled, used, or stored.
* Wear protective work clothing.
* Wash thoroughly immediately after exposure to Arsenic and at the end of the workshift.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Arsenic to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

HEALTH HAZARD INFORMATION

Acute Health Effects

The following acute (short term) health effects may occur immediately or shortly after exposure to Arsenic:

* Skin contact can cause burning, itching and a rash.
* Breathing Arsenic, such as in liquid spray or powder form, can cause nose and throat irritation.
* Eye contact can cause red, watery eyes and irritation.
* High exposures can cause poor appetite, nausea, vomiting and muscle cramps.
* Heart effects with an abnormal EKG can also occur with very high exposures.

Chronic Health Effects

The following chronic (long-term) health effects can occur at some time after exposure to Arsenic and can last for months or years:

Cancer Hazard

* Arsenic is a CARCINOGEN in humans. It has been shown to cause skin and lung cancer.
* Many scientists believe there is no safe level of exposure to a CARCINOGEN. Such substances may also have the potential for causing reproductive damage in humans.

Reproductive Hazard

* Arsenic may damage the developing fetus.
* Arsenic should be handled as a potential teratogenic agent since some Arsenic compounds are known teratogens.

Other Long-Term Effects

* Long-term exposure can cause an ulcer or hole in the "bone" dividing the inner nose. Hoarseness and sore eyes also occur.
* High or repeated exposure can cause nerve damage, with "pins and needles," burning, numbness, and later weakness of arms and legs.
* Repeated skin contact can cause thickened skin and/or patchy areas of darkening and loss of pigment. Some persons develop white lines on the nails.
* Repeated exposure can also damage the liver, cause narrowing of the blood vessels, or interfere with the bone marrow's

ability to make red blood cells.

MEDICAL
Medical Testing
Before first exposure and every 6 to 12 months thereafter, a medical history and exam is recommended, including:

* Exam of the nose, skin, eyes, nails, nervous system.
* Test for urine Arsenic (may not be accurate within 2 days of eating shellfish or fish; most accurate at the end of a workday) should not be greater than 100 micrograms per gram creatinine in the urine.

After suspected overexposure, repeat these tests and consider complete blood count and liver function tests. Also examine your skin periodically for abnormal growths. Skin cancer from Arsenic can be easily cured when detected early.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures
* Arsenic in the presence of acid or acid mist may release a VERY DEADLY gas called Arsine.
* Because smoking can cause heart disease, as well as lung cancer, emphysema, and other respiratory problems, it may worsen respiratory conditions caused by chemical exposure. Even if you have smoked for a long time, stopping now will reduce your risk of developing health problems.

Conditions Made Worse By Exposure
Many scientist believe that skin changes such as thickening and pigment changes make those skin areas more likely to develop skin cancer.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of calc al release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:
* Where possible, automatically transfer Arsenic from drums or other storage containers to process containers.
* Specific engineering controls are recommended for this

chemical by NIOSH. Refer to the NIOSH criteria document: Inorganic Arsenic # 75-149.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Arsenic should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Arsenic.
* If there is the possibility of skin exposure, emergency shower facilities should be provided.
* Wash any areas of the body that may have contacted Arsenic at the end of each workday, whether or not known skin contact has occurred.
* Do not eat, smoke, or drink where Arsenic is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.
* Use a vacuum or a wet method to reduce dust during clean-up. Do not dry sweep.
* When vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Arsenic. Wear protective gloves and clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.

Eye Protection

* Eye protection is included in the recommended respiratory protection.

Respiratory Protection

* IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* At any exposure level, use a MSHA/NIOSH approved supplied-air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self-contained breathing apparatus with a full facepiece operated

in pressure-demand or other positive pressure mode.

HANDLING AND STORAGE

* Prior to working with Arsenic you should be trained on its proper handling and storage.
* Arsenic must be stored to avoid contact with OXIDIZERS (such as PERCHLORATES, PEROXIDES, PERMANGANATES, CHLORATES and NITRATES) and STRONG ACIDS (such as HYDROCHLORIC, SULFURIC and NITRIC) since violent reactions occur.
* A regulated area should be established where Arsenic is handled, used, or stored.
* Store in tightly closed containers in a cool well-ventilated area away from FOOD and COMBUSTIBLE MATERIALS.

Common Name: Arsenic
DOT Number: UN 1558
DOT Emergency Guide code: 53
CAS Number: 7440-38-2

NJ DOH Hazard rating
FLAMMABILITY Not Found
REACTIVITY Not Found

POISONOUS GAS IS PRODUCED IN FIRE
CARCINOGEN

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Use dry chemical, CO2, water spray, or foam extinguishers.
* POISONOUS GAS IS PRODUCED IN FIRE.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES
If Arsenic is spilled, take the following steps:

* Restrict persons not wearing protective equipment from area of spill until clean-up is complete.
* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* It may be necessary to contain and dispose of Arsenic as a HAZARDOUS WASTE. Contact your state Department of Environmental Protection (DEP) or your regional office of the federal Environmental Protection Agency (EPA) for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.
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FIRST AID

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids.

Skin Contact
* Quickly remove contaminated clothing. Immediately wash contaminated skin with large amounts of soap and water.

Antidotes and Special Procedures
* For severe poisoning BAL has been used. For milder poisoning Penicillamine (not penicillin) has been used, both with mixed success. Side effects occur with such treatment and it is NEVER a substitute for controlling exposure. It can only be done under strict medical care.

PHYSICAL DATA

Vapor Pressure: 1 mm Hg at 372oF
Water Solubility: Insoluble

OTHER NAMES AND FORMULATIONS

Arsenicals; Colloidal Arsenic; Metallic Arsenic.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Project
CN 368, Trenton, NJ 08625-0368
(609) 984-2202

ECOLOGICAL INFORMATION

Arsenic is a naturally occurring element which is used to make glass, cloth, and electrical semiconductors. It is also commonly used in fungicides, wood preservatives, growth stimulants for plants and animals, and in veterinary uses. Arsenic enters the environment mainly from its use as a pesticide and from emissions from coal-fueled power plants.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Arsenic metabolism and effects are significantly influenced by the animal/plant tested, the route of administration, the physical and chemical form of the arsenical, and the dose. Inorganic arsenic compounds are more toxic than organic arsenic compounds.

Arsenic has high acute toxicity to aquatic life, birds, and land animals. Except where soil arsenic content is high (around smelters and where arsenic-based pesticides have been used heavily), arsenic does not accumulate in plants to toxic levels. Where soil arsenic content is high, growth and crop yields can be decreased.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Arsenic has high chronic toxicity to aquatic life, and moderate chronic toxicity to birds and land animals.

WATER SOLUBILITY

Arsenic and its salts have low solubility in water. Concentrations of less than 1 milligram will mix with a liter of water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Arsenic is highly persistent in water, with a half-life of more than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of arsenic found in fish tissues is expected to be somewhat higher than the average concentration of arsenic in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA; FWS Biological Rpt 85(1.12); EPA rpt #EPA-450/5-85-002.

Common Name: Cadmium
CAS Number: 7440-43-9
DOT Number: UN 2570
Date: July 31, 1986

HAZARD SUMMARY

* Cadmium can affect you when breathed in.
* Cadmium is a CARCINOGEN, a TERATOGEN, and causes REPRODUCTIVE DAMAGE. HANDLE WITH EXTREME CAUTION.
* High exposures can cause severe lung damage and death. This can be delayed for several hours.
* Repeated lower exposures can cause permanent kidney damage, emphysema, anemia, and/or loss of smell.
* High exposure to Cadmium may cause nausea, salivation, vomiting, cramps, and diarrhea.

IDENTIFICATION

Cadmium is a bluish metal or grayish powder. It is used in electroplating other metals, batteries, pigments, stabilizers for plastics, nuclear reactor rods, and as a catalyst.

REASON FOR CITATION

* Cadmium is on the Hazardous Substance List because it is regulated by OSHA.
* This chemical is also on the Special Health Hazard Substance List because it is a CANCER CAUSING AGENT.
* Definitions are attached.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 0.2 mg/m3 averaged over an 8 hour workshift and 0.6 mg/m3, not to be exceeded during any (15 minute) work period.

NIOSH: It is recommended that exposure to Cadmium be at the lowest feasible level.

ACGIH: The recommended airborne exposure limit is 0.05 mg/m3 averaged over an 8 hour workshift.

* These exposure limits are recommended for Cadmium Dust.
* Cadmium is a PROBABLE CANCER CAUSING AGENT in humans. There may be no safe level of exposure to a carcinogen, so all contact should be reduced to the lowest possible level.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.

* A regulated, marked area should be established where Cadmium is handled, used, or stored.
* Wear protective work clothing.
* Wash thoroughly at the end of the work shift.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Cadmium to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

Metal, metal compounds and alloys are often used in "hot" operations in the workplace. These may include, but are not limited to, welding, brazing, soldering, plating, cutting, and metallizing. At the high temperatures reached in these operations, metals often form metal fumes which have different health effects and exposure standards than the original metal or metal compound and require specialized controls. Your workplace can be evaluated for the presence of particular fumes which may be generated.

HEALTH HAZARD INFORMATION

Acute Health Effects

The following acute (short term) health effects may occur immediately or shortly after exposure to Cadmium:

* During heating or grinding operations, Cadmium can cause a flu like illness with chills, headache, aching and/or fever. This can go on to more serious illness.
* High exposures can cause rapid and severe lung damage, with shortness of breath, chest pain, cough, and even a buildup of fluid in the lungs. In severe cases death or permanent lung damage occurs. Illness can be delayed for 4 to 8 hours, allowing overexposure WITHOUT WARNING. If overexposure is suspected, leave the area; do not wait for signs of illness. Risk is greatest during HEATING and GRINDING operations.
* High exposure to Cadmium may cause nausea, salivation, vomiting, cramps, and diarrhea.

Chronic Health Effects

The following chronic (long term) health effects can occur at some time after exposure to Cadmium and can last for months or years:

Cancer Hazard

* Cadmium (especially Cadmium Oxide) is a PROBABLE CANCER CAUSING AGENT in humans. There is some evidence that it causes prostate and kidney cancer in humans and it has been shown to cause lung and testes cancer in animals.
* Many scientists believe there is no safe level of exposure to a cancer causing agent.

Reproductive Hazard

* It is a PROBABLE TERATOGEN in humans.
* Cadmium may damage the testes (male reproductive glands) and may affect the female reproductive cycle.

Other Long Term Effects

* Repeated low exposures can cause permanent kidney damage which can go unnoticed without testing until severe. The kidney damage can lead to kidney stones and other serious health problems.
* Emphysema and/or lung scarring can occur from a single high exposure or repeated lower exposures.
* Long term exposure can cause anemia, loss of sense of smell, fatigue and/or yellow staining of teeth.

MEDICAL

Medical Testing

Before beginning employment and at regular times after that, the following are recommended:

* Urine test for Cadmium (levels should be less than 10 micrograms per liter of urine).
* Urine test for "low molecular weight proteins" to detect kidney damage
* Urinalysis (UA).
* Lung function tests.

For persons exposed to levels equal to or greater than half the TLV, the following is also recommended:

* Complete blood count (CBC).

These should be repeated after suspected overexposure.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures

* Cigarette smoke contains some Cadmium. Because it is hard for the body to eliminate Cadmium, it tends to build up in the body. Any workplace exposure adds to these levels.
* Smoking or carrying cigarettes near Cadmium increases release of toxic fumes. Also, because both smoking and Cadmium can cause emphysema, lung effects may be greater in smokers.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:

* If Cadmium is used in a "hot" process such as smelting, steel fabricating, or melting Cadmium ingots, Cadmium Fume may be released. This is more acutely toxic than Cadmium Dust and proper controls and protective equipment are necessary.
* Specific engineering controls are recommended for this chemical by NIOSH. Refer to the NIOSH criteria document: Occupational Exposure to Cadmium #76 192.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Cadmium should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Cadmium.
* Wash any areas of the body that may have contacted Cadmium at the end of each workday, whether or not known skin contact has occurred.
* Do not eat, smoke, or drink where Cadmium is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.
* Use a vacuum or a wet method to reduce dust during clean up. DO NOT DRY SWEEP.
* When vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Cadmium. Wear protective gloves and clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.

Eye Protection

* Eye protection is included with the recommended respiratory protection.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* At any exposure level, use a MSHA/NIOSH approved supplied air

respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self contained breathing apparatus with a full facepiece operated in pressure demand or other positive pressure mode.

Common Name: Cadmium
DOT Number: UN 2570
DOT Emergency Guide code: 53
CAS Number: 7440-43-9

NJ DOH Hazard rating

FLAMMABILITY	Not Found
REACTIVITY	Not Found

DO NOT USE WATER
FLAMMABLE POWDER
TOXIC FUMES PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Cadmium is a FLAMMABLE POWDER.
* Toxic Fumes are produced in a fire.
* Use dry chemicals appropriate for extinguishing metal fires. DO NOT USE WATER.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Cadmium is spilled, take the following steps:

* Restrict persons not wearing protective equipment from area of spill until clean up is complete.
* Remove all ignition sources.
* It may be necessary to contain and dispose of Cadmium as a HAZARDOUS WASTE. Contact your state Environmental Program for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.

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HANDLING AND STORAGE

* Prior to working with Cadmium you should be trained on its proper handling and storage.
* A regulated, marked area should be established where Cadmium is handled, used, or stored.
* Cadmium must be stored to avoid contact with SULFUR, SELENIUM, TELLURIUM, AMMONIUM NITRATE, and HYDRAZOIC ACID since violent reactions occur.
* Store in tightly closed containers in a cool well ventilated area away from OXIDIZERS (such as PERCHLORATES, PEROXIDES, PERMANGANATES, CHLORATES, and NITRATES).
* Sources of ignition such as smoking and open flames are prohibited where Cadmium is used, handled, or stored in a manner that could create a potential fire or explosion hazard.

FIRST AID

POISON INFORMATION

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids. Seek medical attention.

Skin Contact
* Remove contaminated clothing. Wash contaminated skin with soap and water.

Breathing
* Remove the person from exposure.
* Begin rescue breathing if breathing has stopped and CPR if heart action has stopped.
* Transfer promptly to a medical facility.
* Medical observation is recommended for 24 to 48 hours after breathing overexposure, as pulmonary edema may be delayed.

PHYSICAL DATA

Water Solubility: Insoluble

OTHER COMMONLY USED NAMES
Chemical Name:
Cadmium

Other Names and Formulations:
C.I. 77180.

ECOLOGICAL INFORMATION

Cadmium is a naturally occurring element used in metal alloys, electroplating, process engraving, photoelectric cells, and in nickel-cadmium electrical storage batteries. Cadmium enters the environment primarily through industrial effluents and landfill leaching.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

In fresh waters, cadmium toxicity is influenced by water hardness--the harder the water, the lower the toxicity. Cadmium has high acute toxicity to aquatic life. No data are available on the short-term effects of cadmium on plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Cadmium has high chronic toxicity to aquatic life. No data are available on the long-term effects of cadmium to plants, birds, or land animals.

WATER SOLUBILITY

Cadmium is slightly soluble in water. Concentrations of less than 1 milligram will mix with a liter of water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Cadmium is highly persistent in water, with a half-life of greater than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of cadmium found in fish tissues is expected to be much higher than the average concentration of cadmium in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA.

Common Name: Copper
CAS Number: 7440-50-8
DOT Number: None
Date: July, 1986

HAZARD SUMMARY

* Copper dust or fumes can affect you when breathed in.
* Eye contact with particles of Copper Metal can cause a severe reaction that can lead to blindness.
* Exposure to dust or fumes can irritate the eyes, nose and throat.
* Copper fumes may cause "metal fume fever". This is a flu-like illness with symptoms of metallic taste, fever and chills, aches, chest tightness and cough.
* Copper may cause an allergic skin rash.

IDENTIFICATION

Copper is a reddish-brown metal. It is widely used in the electrical industry, plumbing, heating, roofing and building construction. It is also used in chemical and pharmaceutical machinery.

REASON FOR CITATION

* Copper is on the Hazardous Substance List because it is regulated by OSHA and cited by ACGIH.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work-related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 1.0 mg/m3 for Copper dusts and mists and 0.1 mg/m3 for Copper Fume averaged over an 8-hour workshift and measured as Copper.

ACGIH: The recommended airborne exposure limit is 1.0 mg/m3 for Copper dusts and mists and 0.2 mg/m3 for Copper fume averaged over an 8-hour workshift and measured as Copper.

* Copper may form metal fumes which present different hazards than the substance itself.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.
* Wear protective work clothing.
* Wash thoroughly immediately after exposure to Copper dust or

fumes.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Copper to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

Metal, metal compounds and alloys are often used in "hot" operations in the work-place. These may include, but are not limited to, welding, brazing, soldering, plating, cutting, and metalizing. At the high temperatures reached in these operations, metals often form metal fumes which have different health effects and exposure standards than the original metal or metal compound and require specialized controls. Your workplace can be evaluated for the presence of particular fumes which may be generated.

HEALTH HAZARD INFORMATION

Acute Health Effects
The following acute (short-term) health effects may occur immediately or shortly after exposure to Copper:

* Eye contact with particles of Copper metal can lead to a severe reaction. This can damage vision and cause blindness.
* Exposure to dust or fumes can irritate the eyes, nose and throat. It may cause coughing and nose bleeds.
* Copper fumes may cause "metal fume fever" with symptoms of metallic taste, chills and fever, aches, cough, and chest tightness. The symptoms may be delayed for several hours after exposure and usually last a day or two.

Chronic Health Effects
The following chronic (long-term) health effects can occur at some time after exposure to Copper and can last for months or years:

Cancer Hazard
* There is evidence that workers in Copper smelters have an increased risk of lung cancer, but this is thought to be due to Arsenic Trioxide exposure and not Copper.

Reproductive Hazard
* According to the information presently available to the New Jersey Department of Health, Copper has been tested and has not been shown to affect reproduction.

Other Long-Term Effects
* Repeated exposure can cause chronic irritation of the nose and may cause ulcers.
* Copper may cause a skin allergy. If allergy develops, very low future exposures can cause itching and a skin rash.
* Repeated exposures can cause thickening of the skin and may cause a greenish color to the skin and hair.
* Repeated, very high Copper exposures can damage the liver.

MEDICAL

Medical Testing
If symptoms develop or overexposure is suspected, the following may be useful:

* Serum and urine Copper levels.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures
Copper metal often contains Arsenic as an impurity. Consult the New Jersey Department of Health Hazardous Substance Fact Sheet on Arsenic if you are exposed to Copper dust or fumes.

Conditions Made Worse By Exposure
"Wilsons Disease" is a rare condition that interferes with the bodies ability to get rid of Copper. If you have this illness, consult your doctor about Copper exposure.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Copper dust or fumes should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Copper dust or fumes.
* Eye wash fountains should be provided in the immediate work area for emergency use.
* Use a vacuum or a wet method to reduce dust during clean-up. Do not dry sweep.
* Do not eat, smoke, or drink where Copper is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.
* On skin contact with Copper dust or fumes, immediately wash or shower to remove the chemical. At the end of the workshift,

wash any areas of the body that may have contacted Copper, whether or not known skin contact has occurred.

* When vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Copper. Wear protective gloves and clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.

Eye Protection

* Wear dustproof goggles when working with powders or dust, unless full facepiece respiratory protection is worn.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* Where the potential exists for exposures over 0.1 mg/m3 as Copper fume or 1 mg/m3 as Copper dusts and mists, use a MSHA/NIOSH approved full facepiece respirator with a high efficiency particulate filter. Greater protection is provided by a powered-air purifying respirator. Particulate filters must be checked every day before work for physical damage, such as rips or tears, and replaced as needed.
* If while wearing a filter, cartridge or canister respirator, you can smell, taste, or otherwise detect Copper, or in the case of a full facepiece respirator you experience eye irritation, leave the area immediately. Check to make sure the respirator-to-face seal is still good. If it is, replace the filter, cartridge, or canister. If the seal is no longer good, you may need a new respirator.
* Be sure to consider all potential exposures in your workplace. You may need a combination of filters, prefilters, cartridges, or canisters to protect against different forms of a chemical (such as vapor and mist) or against a mixture of chemicals.
* Where the potential for high exposures exists, use a MSHA/NIOSH approved supplied-air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

Common Name: Copper
DOT Number: None
DOT Emergency Guide code: No Citation
CAS Number: 7440-50-8

NJ DOH Hazard rating
FLAMMABILITY Not Found
REACTIVITY Not Found

DO NOT USE WATER
TOXIC FUMES ARE PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Use dry chemicals appropriate for extinguishing metal fires. Do not use water.
* TOXIC FUMES ARE PRODUCED IN FIRE.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* It may be necessary to contain and dispose of Copper as a HAZARDOUS WASTE. Contact your state Department of Environmental Protection (DEP) or your regional office of the federal Environmental Protection Agency (EPA) for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.
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HANDLING AND STORAGE

* Prior to working with Copper you should be trained on its proper handling and storage.
* Store in tightly closed containers in a cool well-ventilated area away from ACETYLENE GAS because flammable Hydrogen is produced.
* Copper must be stored to avoid contact with OXIDIZERS such as PERCHLORATES, PEROXIDES, PERMANGANATES, CHLORATES, and NITRATES; CHEMICALLY ACTIVE METALS such as POTASSIUM, SODIUM, MAGNESIUM, and ZINC since violent reactions occur.

FIRST AID
Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids. Consult an ophthalmologist (eye specialist) immediately.

Skin Contact
* Remove contaminated clothing. Wash contaminated skin with water.

PHYSICAL DATA

Water Solubility:
Copper fume: Insoluble
Copper dust and mist: Slightly to Highly soluble.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625-0368

ECOLOGICAL INFORMATION

Copper is a commonly occurring element in our natural water. At low concentrations it is an essential element for both plants and animals. At slightly higher concentrations it is toxic to aquatic life. The toxicity of copper and its compounds to aquatic life varies with the physical and chemical conditions of the water. Factors such as water hardness, alkalinity and pH influence copper toxicity.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Copper and its compounds have high acute toxicity to aquatic life. No data are available on the short-term effects of copper to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Copper and its compounds have high chronic toxicity to aquatic life. No data are available on the long-term effects of copper to plants, birds, or land animals.

WATER SOLUBILITY

Copper and its salts are highly soluble in water. Concentrations of 1,000 milligrams and more will mix with aliter of water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Copper is highly persistent in water, with a half-life greater than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of copper found in fish tissues is expected to be considerably higher than the average concentration of copper in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA.

International Chemical Safety Cards

HYDROGEN SULFIDE

ICSC: 0165

HYDROGEN SULFIDE
Sulfuretted hydrogen
Sulfur hydride
(cylinder)
H_2S
Molecular mass: 34.1

CAS # 7783-06-4
RTECS # MX1225000
ICSC # 0165
UN # 1053
EC # 016-001-00-4

HAZARD SYMBOLS
Consult National Legislation

TYPES OF HAZARD/ EXPOSURE	ACUTE HAZARDS/ SYMPTOMS	PREVENTION	FIRST AID/ FIRE FIGHTING
FIRE	Extremely flammable.	NO open flames, NO sparks, and NO smoking.	Shut off supply; if not possible and no risk to surroundings, let the fire burn itself out; in other cases extinguish with water spray, powder, carbon dioxide.
EXPLOSION	Gas/air mixtures are explosive.	Closed system, ventilation, explosion-proof electrical equipment and lighting. Prevent build-up of electrostatic charges (e.g., by grounding) if in liquid state. Do NOT use compressed air for filling, discharging, or handling.	In case of fire: keep cylinder cool by spraying with water.
EXPOSURE		AVOID ALL CONTACT!	IN ALL CASES CONSULT A DOCTOR!
• **INHALATION**	Cough. Dizziness. Headache. Laboured breathing. Nausea. Sore throat. Unconsciousness. Symptoms may be delayed (see Notes).	Ventilation, local exhaust, or breathing protection.	Fresh air, rest. Half-upright position. Artificial respiration if indicated. Avoid mouth-to-mouth resuscitation. Refer for medical attention.
• **SKIN**	ON CONTACT WITH LIQUID: FROSTBITE.	Cold-insulating gloves.	ON FROSTBITE: rinse with plenty of water, do NOT remove clothes. Refer for medical attention.
• **EYES**	Redness. Pain. Severe deep burns.	Safety goggles, or eye protection in combination with breathing protection.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
• **INGESTION**		Do not eat, drink, or smoke during work.	

SPILLAGE DISPOSAL	STORAGE	PACKAGING & LABELLING
Evacuate danger area! Consult an expert! Ventilation (extra personal protection: self-contained breathing apparatus).	Fireproof. Separated from strong oxidants.	F+ symbol T+ symbol N symbol R: 12-26-50 S: (1/2-)9-16-28-36/37-45-61 UN Hazard Class: 2.3 UN Subsidiary Risks: 2.1

SEE IMPORTANT INFORMATION ON BACK

ICSC: 0165 Prepared in the context of cooperation between the International Programme on Chemical Safety & the Commission of the European Communities © IPCS CEC 1993

International Chemical Safety Cards

HYDROGEN SULFIDE

ICSC: 0165

IMPORTANT DATA

PHYSICAL STATE; APPEARANCE:
COLOURLESS, COMPRESSED LIQUEFIED GAS , WITH CHARACTERISTIC ODOUR OF ROTTEN EGGS.

PHYSICAL DANGERS:
The gas is heavier than air and may travel along the ground; distant ignition possible.

CHEMICAL DANGERS:
Heating may cause violent combustion or explosion. The substance decomposes on burning producing toxic gas (sulfur dioxide - see ICSC # 0074). Reacts violently with strong oxidants, causing fire and explosion hazard. Attacks many metals and some plastic.

OCCUPATIONAL EXPOSURE LIMITS (OELs):
TLV: 10 ppm; 14 mg/m^3 (as TWA); 15 ppm; 21 mg/m^3 (STEL) (ACGIH 1997).

ROUTES OF EXPOSURE:
The substance can be absorbed into the body by inhalation.

INHALATION RISK:
A harmful concentration of this gas in the air will be reached very quickly on loss of containment.

EFFECTS OF SHORT-TERM EXPOSURE:
The substance irritates the eyes and the respiratory tract. Inhalation of gas may cause lung oedema (see Notes). Rapid evaporation of the liquid may cause frostbite. The substance may cause effects on the central nervous system. Exposure may result in unconsciousness. Exposure may result in death. The effects may be delayed. Medical observation is indicated.

EFFECTS OF LONG-TERM OR REPEATED EXPOSURE:

PHYSICAL PROPERTIES

Boiling point: -60°C
Melting point: -85°C
Solubility in water, g/100 ml at 20°C: 0.5
Relative vapour density (air = 1): 1.19

Flash point: Flammable Gas
Auto-ignition temperature: 260°C
Explosive limits, vol% in air: 4.3-46

ENVIRONMENTAL DATA

The substance is very toxic to aquatic organisms.

NOTES

Depending on the degree of exposure, periodic medical examination is indicated. The symptoms of lung oedema often do not become manifest until a few hours have passed and they are aggravated by physical effort. Rest and medical observation are therefore essential. Specific treatment is necessary in case of poisoning with this substance; the appropriate means with instructions must be available. The odour warning when the exposure limit value is exceeded is insufficient.

Transport Emergency Card: TEC (R)-826
NFPA Code: H3; F4; R0;

ADDITIONAL INFORMATION

ICSC: 0165 **HYDROGEN SULFIDE**

Occupational Health Guideline for Hydrogen Sulfide

INTRODUCTION

This guideline is intended as a source of information for employees, employers, physicians, industrial hygienists, and other occupational health professionals who may have a need for such information. It does not attempt to present all data; rather, it presents pertinent information and data in summary form.

SUBSTANCE IDENTIFICATION

- Formula: H_2S
- Synonyms: Sulfuretted hydrogen; hydrosulfuric acid; hepatic gas
- Appearance and odor: Colorless gas with a strong odor of rotten eggs. The odor of this gas should not be used as a warning, since its presence may deaden the sense of smell. Hydrogen sulfide can also exist as a liquid at low temperature and high pressure.

PERMISSIBLE EXPOSURE LIMIT (PEL)

The current OSHA standard for hydrogen sulfide is a ceiling level of 20 parts of hydrogen sulfide per million parts of air (ppm) or a maximum allowable peak of 50 ppm for 10 minutes once, if no other measurable exposure occurs. NIOSH has recommended that the permissible exposure limit be reduced to 15 mg/m³ (10 ppm) averaged over a 10-minute period, and that work areas in which the concentration of hydrogen sulfide exceeds 70 mg/m³ be evacuated. The NIOSH Criteria Document for Hydrogen Sulfide should be consulted for more detailed information.

HEALTH HAZARD INFORMATION

- **Routes of exposure**

Hydrogen sulfide can affect the body if it is inhaled or if it comes in contact with the eyes, skin, nose or throat. It can also affect the body if it is swallowed.

- **Effects of overexposure**

1. Short-term Exposure: Inhalation of high concentrations of hydrogen sulfide vapor may cause loss of consciousness and death. Inhalation of lower concentrations may cause headache, dizziness, and upset stomach. Exposure to hydrogen sulfide can cause temporary loss of the sense of smell, and irritation of the eyes, nose, or throat.

2. Long-term Exposure: Not known.

3. Reporting Signs and Symptoms: A physician should be contacted if anyone develops any signs or symptoms and suspects that they are caused by exposure to hydrogen sulfide.

- **Recommended medical surveillance**

The following medical procedures should be made available to each employee who is exposed to hydrogen sulfide at potentially hazardous levels:

1. Initial Medical Examination:

—A complete history and physical examination: The purpose is to detect pre-existing conditions that might place the exposed employee at increased risk, and to establish a baseline for future health monitoring. Examination of the eyes and lungs should be stressed.

—Eye disease: Hydrogen sulfide is a severe eye irritant and may cause tissue damage. Those with pre-existing eye problems may be at increased risk from exposure.

—14" x 17" chest roentgenogram: Hydrogen sulfide may cause human lung damage. Surveillance of the lungs is indicated.

—FVC and FEV (1 sec): Hydrogen sulfide is a respiratory irritant. Persons with impaired pulmonary function may be at increased risk from exposure. Periodic surveillance is indicated.

2. Periodic Medical Examination: The aforementioned medical examinations should be repeated on an annual basis, except that an x-ray is considered necessary only when indicated by the results of pulmonary function testing, or by signs and symptoms of respiratory disease.

These recommendations reflect good industrial hygiene and medical surveillance practices and their implementation will assist in achieving an effective occupational health program. However, they may not be sufficient to achieve compliance with all requirements of OSHA regulations.

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES
Public Health Service Centers for Disease Control
National Institute for Occupational Safety and Health

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

September 1978

• Summary of toxicology

Hydrogen sulfide gas is a rapidly acting systemic poison which causes respiratory paralysis with consequent asphyxia at high concentrations. It irritates the eyes and respiratory tract at low concentrations. Inhalation of high concentrations of hydrogen sulfide, 1000 to 2000 ppm, may cause coma after a single breath and may be rapidly fatal; convulsions may also occur. Exposure to concentrations of hydrogen sulfide above 50 ppm for one hour may produce acute conjunctivitis with pain, lacrimation, and photophobia; in severe form this may progress to keratoconjunctivitis and vesiculation of the corneal epithelium. In low concentrations, hydrogen sulfide may cause headache, fatigue, irritability, insomnia, and gastrointestinal disturbances; in somewhat higher concentrations it affects the central nervous system, causing excitement and dizziness. Prolonged exposure to 250 ppm of hydrogen sulfide may cause pulmonary edema. Prolonged exposure to concentrations of hydrogen sulfide as low as 50 ppm may cause rhinitis, pharyngitis, bronchitis, and pneumonitis. Repeated exposure to hydrogen sulfide results in increased susceptibility, so that eye irritation, cough, and systemic effects may result from concentrations previously tolerated without any effect. Rapid olfactory fatigue can occur at high concentrations.

CHEMICAL AND PHYSICAL PROPERTIES

• **Physical data**

1. Molecular weight: 34.08
2. Boiling point (760 mm Hg): −60 C (−76 F)
3. Specific gravity (water = 1): Liquid = 1.54
4. Vapor density (air = 1 at 15 C (59 F)): 1.189
5. Melting point: −82.4 C (−116 F)
6. Vapor pressure at 25 C (77 F): 20 atm
7. Solubility in water, g/100 g water at 20 C (68 F): 2.9 (slight)
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Reactivity**

1. Conditions contributing to instability: Elevated temperatures may cause containers to burst.
2. Incompatibilities: Contact with strong oxidizers and oxidizing materials may cause fires and explosions. Hydrogen sulfide attacks many metals, which results in the formation of sulfides.
3. Hazardous decomposition products: Toxic gases and vapors (such as sulfur oxides) may be released in a fire involving hydrogen sulfide.
4. Special precautions: Liquid hydrogen sulfide will attack some forms of plastics, rubber, and coatings.

• **Flammability**

1. Hydrogen sulfide is a flammable gas.
2. Autoignition temperature: 260 C (500 F)
3. Flammable limits in air, % by volume: Lower: 4.3; Upper: 46
4. Extinguishant: Alcohol foam, carbon dioxide

• **Warning properties**

1. Odor Threshold: According to the AIHA *Hygienic Guide*, hydrogen sulfide can be recognized by the "sense of smell at low concentrations. Odor not reliable at high concentrations, and olfactory fatigue occurs quickly . . . . Threshold is 0.13 ppm. Faint but readily perceptible at 0.77 ppm. Easily noticeable at 4.6 ppm. Strong, unpleasant, but not intolerable at 27 ppm." The *Hygienic Guide* also states that "olfactory fatigue can occur with(in) 2 to 15 minutes at 100 ppm."

2. Eye Irritation Level: Grant states that "effects of hydrogen sulfide on the eyes are notable only at sublethal concentrations, most commonly at concentrations so low that they have no discernible systemic effect . . . . Typically, workmen exposed to low concentrations of hydrogen sulfide gas . . . have no sensation of irritation or discomfort for at least several hours, or sometimes for several days while working in the presence of low concentrations. Ocular symptoms generally start after several hours of exposure and may not appear until the patient has finished his work for the day. There is then gradual onset of a scratchy, irritated sensation in the eyes, with tearing and burning . . . . Experimentally it is demonstrable that at a concentration of 100 ppm in air an immediate irritation of the eyes and respiratory tract is produced, but conditions responsible for the vast majority of cases of hydrogen sulfide keratoconjunctivitis are those in which the concentration is too low to cause immediate irritation and has toxic effect only after several hours or days of exposure. However, in industries where the concentration is regularly kept below 10 ppm in air, it is rare to have any irritation of the eyes."

The *Hygienic Guide* states that "50 to 100 ppm causes slight conjunctivitis and respiratory tract irritation after 1 hour."

3. Evaluation of Warning Properties: Since olfactory fatigue occurs at high concentrations, and since the irritant effects are delayed, hydrogen sulfide is treated as a material with poor warning properties.

MONITORING AND MEASUREMENT PROCEDURES

• **Eight-Hour Exposure Evaluation**

Measurements to determine employee exposure are best taken so that the average eight-hour exposure is based on a single eight-hour sample or on two four-hour samples. Several short-time interval samples (up to 30 minutes) may also be used to determine the average exposure level. Air samples should be taken in the employee's breathing zone (air that would most nearly represent that inhaled by the employee).

• **Ceiling Evaluation**

Measurements to determine employee ceiling exposure are best taken during periods of maximum expected airborne concentrations of hydrogen sulfide. Each measurement should consist of a fifteen (15) minute sample or series of consecutive samples totalling fifteen (15)

minutes in the employee's breathing zone (air that would most nearly represent that inhaled by the employee). A minimum of three (3) measurements should be taken on one work shift and the highest of all measurements taken is an estimate of the employee's exposure.

• **Peak Above Ceiling Evaluation**

Measurements to determine employee peak exposure should be taken during periods of maximum expected airborne concentration of hydrogen sulfide. Each measurement should consist of a 10-minute sample or a series of consecutive samples totalling 10 minutes in the employee's breathing zone (air that would most nearly represent that inhaled by the employee). A minimum of three measurements should be taken on one work shift and the highest of all measurements taken is an estimate of the employee's exposure.

• **Method**

Sampling and analyses may be performed by collection of hydrogen sulfide in an impinger containing an alkaline suspension of cadmium hydroxide, followed by chemical treatment, and spectrophotometric analysis. Also, detector tubes certified by NIOSH under 42 CFR Part 84 or other direct-reading devices calibrated to measure hydrogen sulfide may be used. An analytical method for hydrogen sulfide is in the *NIOSH Manual of Analytical Methods*, 2nd Ed., Vol. 6, 1980, available from the Government Printing Office, Washington, D.C. 20402 (GPO No. 017-033-00369-6).

RESPIRATORS

• Good industrial hygiene practices recommend that engineering controls be used to reduce environmental concentrations to the permissible exposure level. However, there are some exceptions where respirators may be used to control exposure. Respirators may be used when engineering and work practice controls are not technically feasible, when such controls are in the process of being installed, or when they fail and need to be supplemented. Respirators may also be used for operations which require entry into tanks or closed vessels, and in emergency situations. If the use of respirators is necessary, the only respirators permitted are those that have been approved by the Mine Safety and Health Administration (formerly Mining Enforcement and Safety Administration) or by the National Institute for Occupational Safety and Health.

• In addition to respirator selection, a complete respiratory protection program should be instituted which includes regular training, maintenance, inspection, cleaning, and evaluation.

PERSONAL PROTECTIVE EQUIPMENT

• Employees should be provided with and required to use impervious clothing, gloves, face shields (eight-inch minimum), and other appropriate protective clothing necessary to prevent the skin from becoming frozen from contact with liquid hydrogen sulfide or from contact with vessels containing liquid hydrogen sulfide.

• Any clothing which becomes wet with liquid hydrogen sulfide should be removed immediately and reworn until the hydrogen sulfide has evapo

• Employees should be provided with and requir use splash-proof safety goggles where liquid hydrogen sulfide may contact the eyes.

COMMON OPERATIONS AND CONTROLS

The following list includes some common operations in which exposure to hydrogen sulfide may occur and control methods which may be effective in each case:

Operation	Controls
Liberation from pockets during underground mining operations near sulfide ores	Local exhaust ventilation; respiratory protective devices
Liberation during refining of high-sulfur petroleum	Concentration and recovery of H_2SO_4
Liberation from accumulations of decaying organic matter in sewers and waste waters of tanneries, glue factories, fat-rendering plants, and fertilizer plants	Provide continuous water discharge to sewer and cover and vent waste drains
Liberation as a by-product of dehairing and tanning process	Provide separate sewage lines and cover and vent waste drains; add neutralizing agents ($CaCl_2$) as appropriate; local exhaust ventilation
Liberation during manufacture of viscose rayon	Local exhaust ventilation
Liberation during production of sulfur dyes, carbon disulfide, sulfur, oleum, and thioprene	Local exhaust ventilation or process enclosure
Liberation during vulcanization of rubber; during manufacture of coke from coal having high gypsum content	Local exhaust ventilation or process enclosure
Liberation during excavation projects	Respiratory protective equipment

Liberation in closed containers containing organic matter	Respiratory protective equipment; life-support line

EMERGENCY FIRST AID PROCEDURES

In the event of an emergency, institute first aid procedures and send for first aid or medical assistance.

• **Eye Exposure**

If liquid hydrogen sulfide gets into the eyes, wash eyes immediately with large amounts of water, lifting the lower and upper lids occasionally. If irritation is present after washing, get medical attention. Contact lenses should not be worn when working with this chemical.

• **Skin Exposure**

If liquid hydrogen sulfide gets on the skin, immediately flush the contaminated skin with water. If liquid hydrogen sulfide penetrates through the clothing, remove the clothing immediately and flush the skin with water. If irritation is present after washing, get medical attention.

• **Breathing**

If a person breathes in large amounts of hydrogen sulfide, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Get medical attention as soon as possible.

• **Rescue**

Move the affected person from the hazardous exposure. If the exposed person has been overcome, notify someone else and put into effect the established emergency rescue procedures. Do not become a casualty. Understand the facility's emergency rescue procedures and know the locations of rescue equipment before the need arises.

SPILL AND LEAK PROCEDURES

• Persons not wearing protective equipment and clothing should be restricted from areas of spills or leaks until cleanup has been completed.

• If hydrogen sulfide is spilled or leaked, the following steps should be taken:

1. Remove all ignition sources.
2. Ventilate area of spill or leak to disperse gas.
3. If in the gaseous form, stop flow of gas. If source of leak is a cylinder and the leak cannot be stopped in place, remove the leaking cylinder to a safe place in the open air, and repair the leak or allow the cylinder to empty.
4. If in the liquid form, allow to vaporize.

REFERENCES

• American Conference of Governmental Industrial Hygienists: "Hydrogen Sulfide," *Documentation of the Threshold Limit Values for Substances in Workroom Air* (3rd ed., 2nd printing), Cincinnati, 1974.

• American Industrial Hygiene Association: "Hydrogen Sulfide," *Hygienic Guide Series*, Detroit, Michigan, 1962.

• Grant, W. M.: *Toxicology of the Eye* (2nd ed.), C. C. Thomas, Springfield, Illinois, 1974.

• Hunter, D.: *Diseases of Occupations* (4th ed.), Little, Brown, Boston, 1969.

• *Hygienic Information Guide No. 20 – Hydrogen Sulfide*, Commonwealth of Pennsylvania, Department of Environmental Resources, Bureau of Occupational Health, 1971.

• Manufacturing Chemists Association, Inc.: *Chemical Safety Data Sheet SD-36, Hydrogen Sulfide*, Washington, D.C., 1968.

• Manufacturing Chemists Association, Inc.: *Chemical Safety Data Sheet SD-71, Phosphorus Pentasulfide*, Washington, D.C., 1958.

• Milby, T. H.: "Hydrogen Sulfide Intoxication – Review of the Literature and Report of Unusual Accident Resulting in Two Cases of Nonfatal Poisoning," *Journal of Occupational Medicine*, 4:431-437, 1962.

• National Institute for Occupational Safety and Health, U.S. Department of Health, Education, and Welfare: *Criteria for a Recommended Standard . . . . Occupational Exposure to Hydrogen Sulfide*, HEW Publication No. (NIOSH) 77-158, GPO No. 017-033-00217-7, U.S. Government Printing Office, Washington, D.C., 1977.

• Patty, F. A. (ed.): *Toxicology*, Vol. II of *Industrial Hygiene and Toxicology* (2nd ed. rev.), Interscience, New York, 1963.

• Sax, N. I.: *Dangerous Properties of Industrial Materials* (3rd ed.), Van Nostrand Reinhold, New York, 1968.

RESPIRATORY PROTECTION FOR HYDROGEN SULFIDE

Condition	Minimum Respiratory Protection* Required Above 10 ppm
Gas Concentration	
300 ppm or less	Any supplied-air respirator with a full facepiece, helmet, or hood. Any self-contained breathing apparatus with a full facepiece.
Greater than 300 ppm or entry and escape from unknown concentrations	Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode. A combination respirator which includes a Type C supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure or continuous-flow mode and an auxiliary self-contained breathing apparatus operated in preasure-demand or other positive pressure mode.
Fire Fighting	Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.
Escape	Any gas mask providing protection against acid gases or hydrogen sulfide. Any escape self-contained breathing apparatus.

*Only NIOSH-approved or MSHA-approved equipment should be used.

Common Name: Lead
CAS Number: 7439-92-1
DOT Number: None
Date: October 1986

HAZARD SUMMARY

* Lead can affect you when breathed in and if swallowed from food, drinks, or cigarettes.
* Lead is a TERATOGEN--HANDLE WITH EXTREME CAUTION.
* Repeated exposure causes Lead build-up in the body. Low levels may cause tiredness, mood changes, headaches, stomach problems and trouble sleeping.
* Higher levels may cause aching, weakness, and concentration or memory problems.
* Lead can also cause serious permanent kidney or brain damage at high levels.
* Lead exposure increases risk of high blood pressure.

IDENTIFICATION

Lead is a heavy, soft gray metal. It has wide industrial use due to its properties of high density, softness, low melting point, resistance to corrosion and ability to stop gamma and x-rays.

REASON FOR CITATION

* Lead is on the RTK Hazardous Substance List because it is regulated by OSHA and cited by ACGIH and NIOSH.
* This chemical is on the Special Health Hazard Substance List because it is a TERATOGEN.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work-related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

* These exposure limits are recommended for inorganic Lead dusts and fumes measured as Lead.

OSHA: The legal airborne permissible exposure limit (PEL) is 0.05 mg/m3 averaged over an 8-hour workshift.

NIOSH: The recommended airborne exposure limit is less than 0.10 mg/m3 averaged over an 10-hour workshift.

ACGIH: The recommended airborne exposure limit is 0.15 mg/m3 averaged over an 8-hour workshift.

* Lead is a TERATOGEN. All contact with this chemical should be reduced to the lowest possible level.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.

* Wear protective work clothing.
* Wash thoroughly at the end of the work-shift.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Lead to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

HEALTH HAZARD INFORMATION

Acute Health Effects
The following acute (short-term) health effects may occur immediately or shortly after exposure to Lead:

* Extremely high exposures could cause seizures, but usually symptoms from Lead occur after weeks to months of exposure.

Chronic Health Effects
The following chronic (long-term) health effects can occur at some time after exposure to Lead and can last for months or years:

Cancer Hazard
* According to the information presently available to the New Jersey Department of Health, Lead has not been tested for its ability to cause cancer in animals.

Reproductive Hazard
* Lead is a PROBABLE TERATOGEN in humans.
* Lead may decrease fertility in males and females.

Other Long-Term Effects
* Repeated exposure to Lead causes Lead to build up in the body. The earliest symptoms may be tiredness, trouble sleeping, stomach problems, constipation, headaches and moodiness (mostly irritability and depression).
* Higher levels may cause aching and weakness in your arms and legs, trouble concentrating and remembering things, and may cause a low blood count (anemia).
* Lead can cause serious, permanent kidney and brain damage at high enough levels.
* Lead exposure increases risk of high blood pressure.

MEDICAL

Medical Testing
Before first exposure and every six months thereafter, OSHA (1910.1025) requires your employer to provide:

* Blood Lead test.
* ZPP test (a special test for the effect of Lead on blood cells).

Before first exposure, and yearly for exposed person with blood Lead over 40 micrograms per 100 g of whole blood, OSHA also

requires a complete medical history and exam with the above tests, and:

* Complete blood count.
* Kidney function tests.

OSHA defines "exposure" for these tests as air levels averages 30 micrograms of Lead or more in a cubic meter of air. OSHA requires your employer to send the doctor a copy of the Lead standard and provide one for you.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures
Body exposures to Lead from hobbies using Lead solder or pigments; target practice; and drinking moonshine made in Leaded containers will increase Lead levels. Repeated breathing or handling Leaded gasoline may also add somewhat to body Lead levels.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.
In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:

* Avoid heating above 900oF.
* Specific engineering controls are required for this chemical by OSHA. Refer to the OSHA standard 1910.1025 available from OSHA or your employer.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* When vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Lead.
* Work clothing should be HEPA vacuumed before removal.
* Do not take contaminated work clothes home. Family members could be exposed.
* Wash any areas of the body that may have contacted Lead at the end of each workday, whether or not known skin contact has occurred.

* Do not eat, smoke, or drink where Lead is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.
* Use a HEPA vacuum or a wet method to reduce dust during clean-up. Do not dry sweep.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Lead dust and fume. Wear protective gloves, full body and hat clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.
* Work clothing should be HEPA vacuumed before removal.

Eye Protection

* Wear dust-proof goggles when working with powders or dust, unless full face-piece respiratory protection is worn.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* Where the potential exists for exposures not higher than 0.5 mg/m3, use a half-mask, air purifying respirator equipped with high efficiency filters.
* Where the potential exists for exposures not higher than 2.5 mg/m3, use a full facepiece, air purifying respirator with high efficiency filters.
* OSHA requires the employer to provide a powered-air purifying respirator, instead of one of the above, whenever the employee asks to use this type of respirator.
* OSHA prohibits the employer from requiring an employee to wear one of the above negative pressure respirators longer than 4.4 hours per day in battery manufacturing and primary and secondary Lead production.
* Where the potential exists for exposures not higher than 50 mg/m3, use any powered-air purifying respirator with high efficiency filters or half-mask supplied-air respirator operated in positive pressure mode.
* If while wearing a filter, cartridge or canister respirator, you can smell, taste or otherwise detect Lead, or in the case of a full facepiece respirator you experience eye irritation, leave the area immediately. Check to make sure the respirator-to-face seal is still good. If it is, replace the filter, cartridge or canister. If the seal is no longer good,

you may need a new respirator.
* Be sure to consider all potential exposures in your workplace. You may need a combination of filters, prefilters, cartridges, or canisters to protect against different forms of a chemical (such as vapor and mist) or against a mixture of chemicals.
* Where the potential exists, for exposures not higher than 100 mg/m3, use supplied-air respirators with full facepiece, hood, helmet or suit, operated in positive pressure mode.
* Where the potential exists for exposures greater than 100 mg/m3, use full facepiece, self-contained breathing apparatus operated in positive pressure mode.

Common Name: Lead
DOT Number: None
DOT Emergency Guide code: No Citation
CAS Number: 7439-92-1

Hazard rating	NFPA
FLAMMABILITY	Not Found
REACTIVITY	Not Found

DO NOT USE WATER
POISONOUS GAS IS PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Lead Powder is FLAMMABLE when exposed to heat or flame.
* POISONOUS GAS IS PRODUCED IN FIRE.
* Use dry chemicals appropriate for extinguishing metal fires. Do not use water.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Lead is spilled, take the following steps:

* Restrict persons not wearing protective equipment from area of spill until clean-up is complete.
* Ventilate the area of spill.
* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* It may be necessary to contain and dispose of Lead as a HAZARDOUS WASTE. Contact your Department of Environmental Protection (DEP) or your regional office of the federal Environmental Protection Agency (EPA) for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.
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HANDLING AND STORAGE

* Prior to working with Lead you should be trained on its proper handling and storage.
* Lead must be stored to avoid contact with OXIDIZERS (such as

PERCHLORATES, PEROXIDES, PERMANGANATES, CHLORATES and NITRATES) and CHEMICALLY ACTIVE METALS (such as POTASSIUM, SODIUM, MAGNESIUM and ZINC) since violent reactions occur.

* Lead is regulated by an OSHA Standard 1910.1025. All requirements of the standard must be followed.

FIRST AID

Eye Contact

* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids.

Skin Contact

* Remove contaminated clothing. Wash contaminated skin with soap and water.

Antidotes and Special Procedures

* Persons with significant Lead poisoning are sometimes treated with EDTA while hospitalized. Since this drug causes a rush of Lead from body organs into the blood and kidneys and thus has its own hazards, it must be done only by experienced medical per sons under careful observation. It or other "chelating" drugs should never be used to prevent poisoning while exposures continues or without strict exposure control as severe kidney damage can result.

PHYSICAL DATA

Vapor Pressure: -1.77 mm Hg at 1832oF
Water Solubility: Slightly soluble

CHEMICAL NAME

Lead

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625-0368

ECOLOGICAL INFORMATION

Lead and its compounds is one of the metals known since ancient times. It occurs widely in the earth's crust and can be dissolved from rocks and minerals into surface waters. Lead and its compounds have a variety of commercial and industrial uses, such as lead pipe, lead-lined containers for corrosive gases and liquids, tetraethyl lead, paint pigments, alloys in metallurgy, storage batteries, ceramics, electronic devices, and plastics.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Toxicity to aquatic life is affected by water hardness - the softer the water, the greater the toxicity. Lead and its compounds have high acute toxicity to aquatic life. Insufficient data are available to evaluate or predict the short-term effects of lead and its compounds to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Lead and its compounds have high chronic toxicity to aquatic life. Lead causes nerve and behavioral effects in humans and could cause similar long-term effects in birds and land animals exposed to lead and its compounds.

WATER SOLUBILITY

Lead and its compounds range in their respective water solubilities from highly soluble to practically insoluble.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Lead and its compounds are highly persistent in water, with a half-life greater than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of lead and its compounds found in fish tissues is expected to be much higher than the average concentration of lead in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA.

Common Name: Mercury
CAS Number: 7439-97-6
DOT Number: UN 2809
Date: October, 1986

HAZARD SUMMARY

* Mercury can affect you when breathed in and by passing through your skin.
* High exposure can cause chest pain, shortness of breath, and a build-up of fluid in the lungs (pulmonary edema). This can cause death.
* Repeated exposures can cause Mercury poisoning with kidney disease, tremors, gum problems, trouble remembering and concentrating and changes in mood.
* Long-term exposure can cause clouding of the eyes.
* Mercury is a corrosive chemical.

IDENTIFICATION

Mercury is a silvery heavy liquid. It is used in thermometers, barometers, vapor lamps, mirror coating, and in making chemicals and electrical equipment.

REASON FOR CITATION

* Mercury is on the RTK Hazardous Substance List because it is cited by OSHA and cited by ACGIH, DOT, NIOSH and DEP.
* This chemical is on the Special Health Hazard Substance List because it is CORROSIVE.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work-related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 0.1 mg/m3, not to be exceeded at any time.
NIOSH: The recommended airborne exposure limit is 0.05 mg/m3 averaged over an 8-hour workshift.
ACGIH: The recommended airborne exposure limit for Mercury Vapor is 0.05 mg/m3, averaged over an 8-hour workshift.

* The above exposure limits are for air levels only. When skin contact also occurs, you may be overexposed, even though air levels are less than the limits listed above.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.
* Wear protective work clothing.
* Wash thoroughly immediately after exposure to Mercury and at

the end of the workshift.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Mercury to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

HEALTH HAZARD INFORMATION

Acute Health Effects
The following acute (short-term) health effects may occur immediately or shortly after exposure to Mercury:

* Exposure to high levels of Mercury vapor can irritate the lungs,causing cough, chest tightness, shortness of breath and fever. This usually begins one to four hours after exposure and can go on to fluid in the lungs (pulmonary edema) and death.

Chronic Health Effects
The following chronic (long-term) health effects can occur at some time after exposure to Mercury and can last for months or years:

Cancer Hazard
* According to the information presently available to the New Jersey Department of Health, Mercury has been tested and has not been shown to cause cancer in animals.

Reproductive Hazard
* There is limited evidence that Mercury may cause an increase in spontaneous abortions in exposed women.
* Organic Mercury substances (organic substances are those which contain carbon) have been identified as human teratogens. While inorganic Mercury substances (those without carbon) have not been shown to be human teratogens, they still should be handled with caution as they may cause reproductive problems in males and females.

Other Long-Term Effects
* Repeated low exposure or a very high single exposure can cause Mercury poisoning. Symptoms include tremors (shaking), trouble remembering and concentrating, gum problems, increased salivation, loss of appetite and weight, and changes in mood and personality. These can be severe and cause hallucinating and psychosis.
* Repeated vapor exposures (usually more than five years) can cause clouding of the eye lens.
* Mercury may cause a skin allergy. If allergy develops, very low future exposures can cause itching and a skin rash.
* Exposure can cause kidney damage.
* Mercury may lower sex drive.

Medical Testing

For those with frequent or potentially high exposure (half the TLV or greater, or significant skin contact), the following are recommended before beginning work and at regular times after that:

* Exam of the nervous system (including handwriting test to detect early hand tremor).
* Urine Mercury level (usually less than 0.02 mg/Liter).
* Kidney function tests.

If symptoms develop or overexposure is suspected, the following may be useful:

* Consider chest x-ray after acute over-exposure.
* Evaluation by a qualified allergist, including careful exposure history and special testing, may help diagnose skin allergy.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:

* Vigorous periodic cleaning of all work surfaces.
* Where possible, automatically pump liquid Mercury from drums or other storage containers to process containers.
* Specific engineering controls are recommended for this chemical by NIOSH. Refer to the NIOSH criteria document: Occupational Exposure to Mercury #73-11024.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Mercury should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Mercury.
* On skin contact with Mercury, immediately wash or shower to

health. If the possibility of exposures above 28 mg/m3 exists use a MSHA/NIOSH approved self-contained breathing apparatus with a full facepiece operated in continuous flow or other positive pressure mode.

Common Name: Mercury
DOT Number: UN 2809
DOT Emergency Guide code: 60
CAS Number: 7439-97-6

NJ DOH Hazard rating
FLAMMABILITY Not Found
REACTIVITY Not Found

POISONOUS GAS IS PRODUCED IN FIRE
CORROSIVE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Mercury may burn, but does not readily ignite.
* Use dry chemical, CO2, water spray, or foam extinguishers.
* POISONOUS GAS IS PRODUCED IN FIRE.
* Use water to keep fire exposed containers cool.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Mercury is spilled or leaked, take the following steps:

* Restrict persons not wearing protective equipment from area of spill or leak until clean-up is complete.
* Spills should be collected with special Mercury vapor suppressants or special vacuums. Kits specific for clean-up of Mercury spills are available.
* It may be necessary to contain and dispose of Mercury as a HAZARDOUS WASTE. Contact your Department of Environmental Protection (DEP) or your regional office of the federal Environmental Protection Agency (EPA) for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.

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HANDLING AND STORAGE

* Prior to working with Mercury you should be trained on its proper handling and storage.
* Mercury must be stored to avoid contact with CHLORINE DIOXIDE, NITRIC ACID, NITRATES, ETHYLENE OXIDE, CHLORINE and METHYLAZIDE since violent reactions occur.
* Store in tightly closed containers in a cool, well-ventilated area away from ACETYLENE, AMMONIA and NICKEL.
* Mercury may initiate fires of other combustible materials.

FIRST AID

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids.

Skin Contact
* Quickly remove contaminated clothing. Immediately wash contaminated skin with large amounts of water.

Breathing
* Remove the person from exposure.
* Begin rescue breathing if breathing has stopped and CPR if heart action has stopped.
* Transfer promptly to a medical facility.
* Medical observation is recommended for 24 to 48 hours after breathing overexposure, as pulmonary edema may be delayed.

PHYSICAL DATA

Vapor Pressure: 0.0012 mm Hg at 68oF
Water Solubility: Insoluble

Other Names and Formulations:
Colloidal Mercury; Quick Silver.

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ECOLOGICAL INFORMATION

Elemental mercury is a heavy and relatively inert liquid which is oxidized to inorganic mercury (II) under natural conditions. Mercury (II) may combine with an organic fraction to from methylmercury. Both mercury (II) and methylmercury are of environmental concern. Mercury (II) may enter the environment in industrial or municipal waste treatment discharges, from previously contaminated sediments, and from the weathering of natural rocks. Bacteria may then convert it into methylmercury. The concentration of mercury (II) in bodies of water may be elevated with acid rain due to the scouring of mercury from the air and increased partitioning from the sediment into the water.

ACUTE (SHORT TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Mercury(II) and methylmercury has high acute toxicity to aquatic life. Insufficient data are available to evaluate or predict the short term effects of mercury (II) or methylmercury to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive

problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Mercury (II) and methylmercury have high chronic toxicity to aquatic life. Eating fish contaminated with mercury residues has caused secondary poisoning in humans: birds or land animals similarly exposed to mercury and its compounds could also be subject to such effects. Insufficient data are available to evaluate or predict the long-term effects of mercury and its compounds to plants.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Mercury is highly persistent in water, with a half-life greater than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.
The concentration of mercury(II) and methylmercury found in fish tissues is expected to be considerably higher than the average concentration of mercury(II) or methylmercury in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL, Duluth, U.S.EPA, Phytotox.

Common Name: Methyl Alcohol
CAS Number: 67-56-1
DOT Number: UN 1230
Date: October 30, 1986

HAZARD SUMMARY

* Methyl Alcohol can affect you when breathed in and by passing through your skin.
* Exposure can cause blindness.
* It may damage the liver.
* Exposure to high concentrations can cause headaches, nausea, vomiting and dizziness. It can cause death.
* Repeated or prolonged contact can cause dryness and cracking of the skin.
* Methyl Alcohol is a FLAMMABLE LIQUID and a FIRE HAZARD.

IDENTIFICATION

Methyl Alcohol is a colorless liquid with a strong odor. It is used as a solvent and cleaner.

REASON FOR CITATION

* Methyl Alcohol is on the RTK Hazardous Substance List because it is regulated by OSHA and cited by ACGIH, DOT, NIOSH and NFPA.
* This chemical is on the Special Health Hazard Substance List because it is FLAMMABLE.
* Definitions are attached.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.
* ODOR THRESHOLD = 100 ppm.
* The odor threshold only serves as a warning of exposure. Not smelling it does not mean you are not being exposed.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 200 ppm averaged over an 8 hour workshift.

NIOSH: The recommended airborne exposure limit is 200 ppm averaged over an 10 hour workshift and 800 ppm, not to be exceeded during any 15 minute work period.

ACGIH: The recommended airborne exposure limit is 200 ppm averaged over an 8 hour workshift and 250 ppm as a STEL (short term exposure limit).

* The above exposure limits are for air levels only. When skin contact also occurs, you may be overexposed, even though air levels are less than the limits listed above.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust

symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:

* Where possible, automatically pump liquid Methyl Alcohol from drums or other storage containers to process containers.
* Specific engineering controls are recommended for this chemical by NIOSH. Refer to the NIOSH criteria document on Methyl Alcohol # 76 148.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Methyl Alcohol should change into clean clothing promptly.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Methyl Alcohol.
* If there is the possibility of skin exposure, emergency shower facilities should be provided.
* Wash any areas of the body that may have contacted Methyl Alcohol at the end of each work day, whether or not known skin contact has occurred.
* Do not eat, smoke, or drink where Methyl Alcohol is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Methyl Alcohol. Wear solvent resistant gloves and clothing. Safety equipment suppliers/ manufacturers can provide recommendations on the most protective glove/ clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.
* ACGIH recommends Nitrile Rubber or VITON as good to excellent protective materials.

Eye Protection

* Wear splash proof chemical goggles and face shield when working with liquid, unless full facepiece respiratory protection is worn.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* Where the potential exists for exposures over 200 ppm, use an MSHA/NIOSH approved supplied air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use an MSHA/NIOSH approved self contained breathing apparatus with a full facepiece operated in pressure demand or other positive pressure mode.
* Exposure to 25,000 ppm is immediately dangerous to life and health. If the possibility of exposures above 25,000 ppm exists, use a MSHA/NIOSH approved self contained breathing apparatus with a full facepiece operated in continuous flow or other positive pressure mode.

HANDLING AND STORAGE

* Prior to working with Methyl Alcohol you should be trained on its proper handling and storage.
* Methyl Alcohol must be stored to avoid contact with STRONG OXIDIZERS (such as CHLORINE, BROMINE, and FLUORINE).
* Store in tightly closed containers in a cool well ventilated area away from HEAT.
* Sources of ignition such as smoking and open flames are prohibited where Methyl Alcohol is handled, used, or stored.
* Metal containers involving the transfer of 5 gallons or more should be grounded and bonded. Drums must be equipped with self closing valves, pressure vacuum bungs, and flame arresters.
* Use only non sparking tools and equipment, especially when opening and closing containers of Methyl Alcohol.

Common Name: Methyl Alcohol
DOT Number: UN 1230
DOT Emergency Guide code: 28
CAS Number: 67-56-1

NJ DOH Hazard rating

FLAMMABILITY	3
REACTIVITY	0

FLAMMABLE LIQUID
POISONOUS GASES ARE PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Methyl Alcohol is a FLAMMABLE LIQUID.
* Use dry chemical, CO2, or alcohol foam extinguishers and water to keep fire exposed containers cool.
* POISONOUS GASES ARE PRODUCED IN FIRE, including Formaldehyde.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Methyl Alcohol is spilled or leaked, take the following steps:

* Restrict persons not wearing protective equipment from area of spill or leak until cleanup is complete.
* Remove all ignition sources.
* Ventilate area of spill or leak.
* Absorb liquids in vermiculite, dry sand, earth, or a similar material and deposit in sealed containers.
* Keep Methyl Alcohol out of a confined space, such as a sewer, because of the possibility of an explosion, unless the sewer is designed to prevent the buildup of explosive concentrations.
* It may be necessary to contain and dispose of Methyl Alcohol as a HAZARDOUS WASTE. Contact your state Environmental Program for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.

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FIRST AID

POISON INFORMATION

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids. Seek medical attention immediately.

Skin Contact
* Quickly remove contaminated clothing. Immediately wash area with large amounts of water. Seek medical attention.

Breathing
* Remove the person from exposure.
* Begin rescue breathing if breathing has stopped and CPR if heart action has stopped.
* Transfer promptly to a medical facility.

PHYSICAL DATA

Vapor Pressure: 97 mm Hg at 68oF

Flash Point: 52oF
Water Solubility: Miscible

OTHER COMMONLY USED NAMES

Chemical Name:
Methanol

Other Names and Formulations:
Wood Alcohol; Carbinol; Methylol.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625 0368

ECOLOGICAL INFORMATION

Methyl Alcohol is a clear, colorless liquid with a mild odor and is one of the largest commodity chemicals in the world. It is used mainly as a feedstock to make other chemicals, but also has potential markets as a fuel and to make animal feed additives. It may enter the environment from industrial discharges or from spills.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Methyl Alcohol has slight acute toxicity to aquatic life. It has caused germination and size decrease and other injury to agricultural and ornamental crops. Insufficient data are available to evaluate or predict the short-term effects of methanol to birds or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Methyl Alcohol has slight chronic toxicity to aquatic life. Insufficient data are available to evaluate or predict the long-term effects of methanol to plants, birds, or land animals.

WATER SOLUBILITY

Methyl Alcohol is highly soluble in water. Concentrations of 1,000 milligrams and more will mix with a liter of water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Methyl Alcohol is slightly persistent in water, with a half-life of

between 2 to 20 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded. About 86.5% of Methyl Alcohol will eventually end up in water; the rest will end up in the air.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of Methyl Alcohol found in fish tissues is expected to be about the same as the average concentration of Methyl Alcohol in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA., Phytotox.

Common Name: Nickel
CAS Number: 7440-02-0
DOT Number: UN 2881 (Nickel catalyst, dry)
Date: April, 1989

HAZARD SUMMARY

* Nickel dusts and fumes can affect you when breathed in.
* Nickel is a CARCINOGEN and may damage the developing fetus. HANDLE WITH EXTREME CAUTION. Cancers in humans are associated with Nickel refining.
* Skin contact may cause skin allergy, with itching, redness and later rash.
* Lung allergy occasionally occurs with asthma-type effects.
* High exposure can cause cough, shortness of breath and fluid in the lungs, which is sometimes delayed for 1 to 2 days after exposure.
* It is a HIGHLY FLAMMABLE SOLID and is a DANGEROUS FIRE and EXPLOSION HAZARD.

IDENTIFICATION

Nickel is a silvery-white metal. It is used in electroplating and in making coins, batteries, catalysts and metal alloys such as stainless steel.

REASON FOR CITATION

* Nickel is on the Hazardous Substance List because it is regulated by OSHA and cited by ACGIH, DOT, NIOSH, IARC, NTP, DEP, NFPA and EPA.
* It is on the Special Health Hazard Substance List because it is a CARCINOGEN.

HOW TO DETERMINE IF YOU ARE BEING exposed

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work-related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 1 mg/m3 averaged over an 8-hour workshift. (Final Rule January 1989).

NIOSH: The recommended airborne exposure limit is 0.015 mg/m3 averaged over a 10-hour workshift.

ACGIH: The recommended airborne exposure limit is 1 mg/m3 averaged over an 8-hour workshift.

* Nickel may form metal fumes which present different hazards than the substance itself.
* Nickel is a PROBABLE CARCINOGEN in humans. There may be no safe level of exposure to a carcinogen, so all contact should be reduced to the lowest possible level.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust

ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.
* A regulated, marked area should be established where Nickel is handled, used, or stored.
* Wear protective work clothing.
* Wash thoroughly immediately after exposure to Nickel.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Nickel to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

Metal, metal compounds and alloys are often used in "hot" operations in the work-place. These may include, but are not limited to, welding, brazing, soldering, plating, cutting, and metallizing. At the high temperatures reached in these operations, metals often form metal fumes which have different health effects and exposure standards than the original metal or metal compound and require specialized controls. Your workplace can be evaluated for the presence of particular fumes which may be generated.

HEALTH HAZARD INFORMATION

Acute Health Effects

The following acute (short-term) health effects may occur immediately or shortly after exposure to Nickel:

* Eye or skin contact may cause irritation.
* Fumes from heated Nickel can cause a pneumonia-like illness, with cough and shortness of breath. Higher exposures can cause a build-up of fluid in the lungs (pulmonary edema), a medical emergency, with severe shortness of breath.

Chronic Health Effects

The following chronic (long-term) health effects can occur at some time after exposure to Nickel and can last for months or years:

Cancer Hazard

* Nickel is a PROBABLE CARCINOGEN in humans. There is some evidence that it causes lung and nasal sinus cancer in humans and it has been shown to cause lung cancer in animals.
* There is a clear association between Nickel refining and an increase in lung, nasal and throat cancers in humans.
* Many scientists believe there is no safe level of exposure to a carcinogen. Such substances may also have the potential for causing reproductive damage in humans.

Reproductive Hazard

* Nickel may damage the developing fetus.

Other Long-Term Effects

* Skin contact can cause allergy. Symptoms include burning, itching, redness and bumps or other rash. Rash may spread to

other areas and last for weeks after exposure stops, but usually improves in about a week.
* Lung allergy (asthma) occasionally occurs, with wheezing and/or tightness in the chest.
* Exposure to Nickel can cause a sore or hole in the "bone" dividing the inner nose (septum).
* Single high or repeated lower exposures may damage the lungs, with scarring of lung tissues, and may cause damage to heart muscle, liver and/or kidney.

MEDICAL

Medical Testing
Before beginning employment and at regular times after that, the following are recommended:

* Lung function tests. These may be normal if the person is not having an attack at the time of the test.
* Urine or plasma test for Nickel (unexposed persons have urine levels less than 10 micrograms per liter).

If symptoms develop or overexposure is suspected, the following may be useful:

* Daily urine Nickel for several days (persons with urine Nickel over 100 micrograms per liter need medical attention).
* Consider chest x-ray after acute over-exposure.
* Evaluation by a qualified allergist, including careful exposure history and special testing, may help diagnose skin allergy.
* Liver and kidney function tests.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures
Because smoking can cause heart disease, as well as lung cancer, emphysema, and other respiratory problems, it may worsen respiratory conditions caused by chemical exposure. Even if you have smoked for a long time, stopping now will reduce your risk of developing health problems.

Conditions Made Worse By Exposure
Persons who are allergic to Nickel may also react to Nickel-coated jewelry, watchbands and, sometimes, to prolonged contact with keys, coins, etc.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1) how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following control is recommended:

* Specific engineering controls are recommended for this chemical by NIOSH. Refer to the NIOSH criteria document: Occupational Exposure to Inorganic Nickel #77-164.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Nickel should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Nickel.
* Wash any areas of the body that may have contacted Mercuric Sulfate at the end of each workday, whether or not known skin contact has occurred.
* Do not eat, smoke, or drink where Nickel is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.
* Use a vacuum or a wet method to reduce dust during clean-up. Do not dry sweep.
* When vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Nickel. Wear protective gloves and clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.

Eye Protection

* Eye protection is included in the recommended respiratory protection.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training,

respirator fit testing and medical exams, as described in OSHA 1910.134.

* At any exposure level, use a MSHA/NIOSH approved supplied-air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

HANDLING AND STORAGE

* Prior to working with Nickel you should be trained on its proper handling and storage.
* A regulated, marked area should be established where Nickel is handled, used, or stored.
* Nickel must be stored to avoid contact with STRONG ACIDS (such as HYDROCHLORIC, SULFURIC and NITRIC) since violent reactions occur.
* Store in tightly closed containers in a cool, well-ventilated area away from ACIDS, FLUORINE, AMMONIA, PHOSPHORUS, SULFUR, SELENIUM, HYDRAZINE and PERFORMIC ACID.
* Sources of ignition, such as smoking and open flames, are prohibited where Nickel is used, handled, or stored in a manner that could create a potential fire or explosion hazard.
* Wherever Nickel is used, handled, manufactured, or stored, use explosion-proof electrical equipment.

Common Name: Nickel
DOT Number: UN 2881 Nickel catalyst, dry
DOT Emergency Guide code: 53
CAS Number: 7440-02-0

Hazard rating	NFPA
FLAMMABILITY	4
REACTIVITY	0

FLAMMABLE WHEN IN DUST FORM
CARCINOGEN
POISONOUS GASES ARE PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Nickel dust is FLAMMABLE.
* Use dry chemical, soda ash, or lime extinguishers.
* POISONOUS GASES ARE PRODUCED IN FIRE, including Nickel Carbonyl.
* Dry Nickel catalyst may spontaneously ignite and the fire may restart after it has been extinguished.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Nickel is spilled, take the following steps:

* Restrict persons not wearing protective equipment from area of

spill until clean-up is complete.
* Remove all ignition sources.
* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* Keep Mercuric Sulfate out of a confined space, such as a sewer, because of the possibility of an explosion, unless the sewer is designed to prevent the build-up of explosive concentrations.
* It may be necessary to contain and dispose of Nickel as a HAZARDOUS WASTE. Contact your Department of Environmental Protection (DEP) or your regional office of the federal Environmental Protection Agency (EPA) for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.
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FIRST AID
Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids.

Skin Contact
* Remove contaminated clothing. Wash contaminated skin with water.

Breathing
* Remove the person from exposure.
* Transfer promptly to a medical facility.
* Begin rescue breathing if breathing has stopped and CPR if heart action has stopped.
* Medical observation and tests for urine Nickel are recommended for 24 to 48 hours after breathing overexposure, as pulmonary edema may be delayed.

PHYSICAL DATA

Vapor Pressure: Essentially zero mm Hg at 68oF (20oC)
Water Solubility: Insoluble

OTHER COMMONLY USED NAMES

Chemical Name:
Nickel

Other Names and Formulations:
Raney Alloy, C.I. 77775.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625-0368

ECOLOGICAL INFORMATION

Nickel is one of the most common metals occurring in surface

waters. It occurs naturally in surface waters from the weathering of rocks. Other sources of nickel and compounds to the environment include the burning of coal and other fossil fuels and discharges from such industries as electroplating and smelting.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Water hardness affects nickel toxicity to aquatic organisms -the softer the water, the higher the toxicity.

Nickel and its compounds have high acute toxicity to aquatic life. Insufficient data are available to evaluate or predict the short-term effects of nickel and its compounds to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Nickel and its compounds have high chronic toxicity to aquatic life. Insufficient data are available to evaluate or predict the long-term effects of nickel and its compounds to plants, birds, or land animals.

WATER SOLUBILITY

Nickel and its compounds have water solubilities ranging from low to high.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Nickel and its compounds are highly persistent in water, with half-lives greater than 200 days.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.
The concentration of nickel and its compounds found in fish tissues is expected to be somewhat higher than the average concentration of nickel and its compounds in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA.

Please reduce your browser font size for better viewing and printing.

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

NITRIC ACID, 50-70%

MSDS Number: N3660 --- *Effective Date: 12/08/96*

1. Product Identification

Synonyms: Aqua Fortis; Azotic Acid; Nitric Acid 50%; Nitric Acid 65%; nitric acid 69-70%
CAS No.: 7697-37-2
Molecular Weight: 63.00
Chemical Formula: HNO3
Product Codes: J.T. Baker: 5371, 5555, 5876, 9597, 9598, 9600, 9601, 9602, 9604, 9606, 9607, 9616 Mallinckrodt: 1409, 2703, 2704, 6623, V069, V077, V336, V561

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Nitric Acid	7697-37-2	65 - 70%	Yes
Water	7732-18-5	30 - 35%	No

3. Hazards Identification

Emergency Overview

POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Yellow (Reactive)

Potential Health Effects

Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

Inhalation:
Corrosive! Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract.

Ingestion:
Corrosive! Swallowing nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract.

Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color.

Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth and lung damage. Long-term exposures seldom occur due to the corrosive properties of the acid.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye disease, or cardiopulmonary diseases may be more susceptible to the effects of this substance.

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4. First Aid Measures

Immediate first aid treatment reduces the health effects of this substance.
Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult,

give oxygen. Call a physician.

Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

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5. Fire Fighting Measures

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.

Explosion:
Reacts explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc. Reacts with most metals to release hydrogen gas which can form explosive mixtures with air.

Fire Extinguishing Media:
Water spray may be used to keep fire exposed containers cool. Do not get water inside container.

Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

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6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such

as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802. J. T. Baker NEUTRASORB(tm) or TEAM(tm) 'Low Na+' acid neutralizers are recommended for spills of this product.

7. Handling and Storage

Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it for other purposes. When diluting, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
-OSHA Permissible Exposure Limit (PEL): 2 ppm (TWA), 4 ppm (STEL) -ACGIH Threshold Limit Value (TLV): 2 ppm (TWA); 4 ppm (STEL)

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Nitric acid is an oxidizer and should not come in contact with cartridges and canisters that contain oxidizable materials, such as activated charcoal. Canister-type respirators using sorbents are ineffective.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Colorless to yellowish liquid.

Odor:
Suffocating, acrid.

Solubility:
Infinitely soluble.

Specific Gravity:
1.41

pH:
1.0 (0.1M solution)

% Volatiles by volume @ 21C (70F):
100 (as water and acid)

Boiling Point:
122C (252F)

Melting Point:
-42C (-44F)

Vapor Density (Air=1):
2-3

Vapor Pressure (mm Hg):
48 @ 20C (68F)

Evaporation Rate (BuAc=1):
No information found.

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.

Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate. Will react with water or steam to produce heat and toxic and corrosive fumes.

Hazardous Polymerization:
Will not occur.

Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.

Conditions to Avoid:
Light and heat.

11. Toxicological Information

Nitric acid: Inhalation rat LC50: 244 ppm (NO2)/30M; Investigated as a mutagen, reproductive effector. Oral (human) LDLo: 430 mg/kg.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Nitric Acid (7697-37-2)	No	No	None
Water (7732-18-5)	No	No	None

12. Ecological Information

Environmental Fate:
No information found.

Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste facility. Although not a listed RCRA hazardous waste, this material may exhibit one or more characteristics of a hazardous waste and require appropriate analysis to determine specific disposal requirements. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 150LB

International (Water, I.M.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 150LB

International (Air, I.C.A.O.)

Proper Shipping Name: NITRIC ACID
Hazard Class: 8
UN/NA: UN2031
Packing Group: I
Information reported for product/size: 150LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Nitric Acid (7697-37-2)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Nitric Acid (7697-37-2)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\---------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Nitric Acid (7697-37-2)	1000	1000	Yes	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\---------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)

Nitric Acid (7697-37-2)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6

WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: 3 **Flammability:** 0 **Reactivity:** 0 **Other:** Oxidizer

Label Hazard Warning:
POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

Label Precautions:
Do not get in eyes, on skin, or on clothing. Do not breathe vapor or mist. Use only with adequate ventilation. Wash thoroughly after handling. Keep from contact with clothing and other combustible materials. Do not store near combustible materials. Store in a tightly closed container. Remove and wash contaminated clothing promptly.

Label First Aid:
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases get medical attention immediately.

Product Use:
Laboratory Reagent.

Revision Information:
Mixture. New 16 section MSDS format, all sections have been revised.

Disclaimer:
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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

Common Name: Zinc
CAS Number: 7440-66-6
DOT Number: UN 1436
Date: January 31, 1986

HAZARD SUMMARY

* Zinc can affect you when breathed in.
* Zinc dust particles can irritate the eyes.
* Exposure to solid Zinc is not known to cause acute or chronic health effects, but heated Zinc may give off Zinc Oxide Fume which can cause health effects.
* Metal fragments can scratch the eyes.
* When Zinc is refined, Cadmium is released. Cadmium is a cancer causing agent.

IDENTIFICATION

Zinc is a soft white metal with a bluish tinge. It is used as a coating on iron and steel, and in making brass metal alloys, also as a dust in making paint and dyestuffs.

REASON FOR CITATION

* Zinc is on the Hazardous Substance List because it is cited by DOT, NFPA and EPA.
* Definitions are attached.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

* No exposure limits have been established for Zinc. Metal, metal compounds and alloys are often used in "hot" operations in the workplace. These may include, but are not limited to, welding, brazing, soldering, plating, cutting, and metallizing. At the high temperatures reached in these operations, metals often form metal fumes which have different health effects and exposure standards than the original metal or metal compound and require specialized controls. Your workplace can be evaluated for the presence of particular fumes which may be generated.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.
* Wear protective work clothing.
* Wash thoroughly at the end of the workshift.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Zinc to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

HEALTH HAZARD INFORMATION

Acute Health Effects

The following acute (short term) health effects may occur immediately or shortly after exposure to Zinc:

* Metal particles can irritate the eyes.
* High exposure to Zinc dust, like any dust, can cause cough with phlegm.

Chronic Health Effects

The following chronic (long term) health effects can occur at some time after exposure to Zinc and can last for months or years:

Cancer Hazard

* According to the information presently available to the New Jersey Department of Health, Zinc has not been tested for its ability to cause cancer in animals.

Reproductive Hazard

* According to the information presently available to the New Jersey Department of Health, Zinc has not been tested for its ability to adversely affect reproduction.

Other Long Term Effects

* Zinc has not been tested for other chronic (long term) health effects.

MEDICAL

Medical Testing

There is no special test for this chemical. However, if illness occurs or over exposure is suspected, medical attention is recommended.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

In evaluating the controls present in your workplace, consider: (1)

how hazardous the substance is, (2) how much of the substance is released into the workplace and (3) whether harmful skin or eye contact could occur. Special controls should be in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Zinc dust should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to Zinc dust.
* Wash any areas of the body that may have contacted Zinc dust at the end of each workday, whether or not known skin contact has occurred.
* Use a vacuum or a wet method to reduce dust during cleanup. DO NOT DRY SWEEP.
* Do not eat, smoke, or drink where Zinc dust is handled, processed, or stored, since the chemical can be swallowed. Wash hands carefully before eating or smoking.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing
* Avoid skin contact with Zinc dust. Wear protective gloves and clothing. Safety equipment suppliers/manufacturers can provide recommendations on the most protective glove/clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.

Eye Protection
* Wear dust proof goggles when working with powders or dust, unless full face piece respiratory protection is worn.

Respiratory Protection
IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* Where the potential exists for exposures to Zinc dusts, use a MSHA/NIOSH approved respirator equipped with particulate (dust/fume/mist) filters. Particulate filters must be checked every day before work for physical damage, such as rips or tears, and replaced as needed.

* For processes where Zinc is heated refer to respiratory protection recommendations on the NJ Hazardous Substance Fact Sheet on Zinc Oxide.
* If while wearing a filter, cartridge or canister respirator, you can smell, taste, or otherwise detect Zinc, or in the case of a full facepiece respirator you experience eye irritation, leave the area immediately. Check to make sure the respirator to face seal is still good. If it is, replace the filter, cartridge, or canister. If the seal is no longer good, you may need a new respirator.
* Be sure to consider all potential exposures in your workplace. You may need a combination of filters, prefilters, cartridges, or canisters to protect against different forms of a chemical (such as vapor and mist) or against a mixture of chemicals.
* Where the potential for high exposures exists, use a MSHA/NIOSH approved supplied air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self contained breathing apparatus with a full facepiece operated in pressure demand or other positive pressure mode.

Common Name: Zinc
DOT Number: UN 1436
DOT Emergency Guide code: 76
CAS Number: 7440-66-6

NJ DOH Hazard rating

FLAMMABILITY	1
REACTIVITY	1

DUST FORMS EXPLOSIVE MIXTURE WITH AIR
COMBUSTIBLE DUSTS
IRRITATING & POISONOUS GASES/FUMES PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Zinc is a COMBUSTIBLE SOLID.
* Use dry chemical, sand, or foam extinguishers.
* POISONOUS GAS IS PRODUCED IN FIRE.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Zinc is spilled, take the following steps:

* Restrict persons not wearing protective equipment from area of spill until cleanup is complete.
* Remove all ignition sources.
* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* It may be necessary to contain and dispose of Zinc as a HAZARDOUS WASTE. Contact your state Environmental Pro gram for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.
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HANDLING AND STORAGE

* Prior to working with Zinc you should be trained on its proper handling and storage.
* Zinc must be stored to avoid contact with CHROMIC ANHYDRIDE, MANGANESE CHLORIDE, CHLORATES, CHLORINE and MAGNESIUM since violent reactions occur.
* Store in tightly closed containers in a cool well ventilated area away from WATER, ACIDS and ALKALI HYDROXIDES, because flammable Hydrogen gas is produced.
* Sources of ignition such as smoking and open flames are prohibited where Zinc is used, handled, or stored in a manner that could create a potential fire or explosion hazard.

FIRST AID

POISON INFORMATION

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids. Seek medical attention immediately.

Skin Contact
* Remove contaminated clothing. Wash contaminated skin with soap and water.

Breathing
* Remove the person from exposure.

PHYSICAL DATA

Water Solubility: Insoluble

Other Names and Formulations:
Blue Powder; Granular Zinc; Emanay Zinc Dust.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625 0368

ECOLOGICAL INFORMATION

Zinc is one of the most commonly used metals in the world. Its major uses are for galvanizing steel, producing alloys, and for serving as an ingredient in rubber and paints. Zinc is an essential element for life when present in trace amounts. Zinc exists as a variety of salts. Zinc may enter the environment in the discharge from galvanizing plants, as a leachate from galvanized structures and natural ores, and from municipal waste treatment plant discharges.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

The toxicity of zinc to aquatic life is related to water hardness, with increased toxicity occurring in softer waters. Zinc and its salts have high acute toxicity to aquatic life. Insufficient data are available to evaluate or predict the short-term effects of zinc and its compounds to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Zinc and its salts have high chronic toxicity to aquatic life. Insufficient data are available to evaluate or predict the long-term effects of zinc and its salts to plants, birds, or land animals.

WATER SOLUBILITY

Zinc exists as a variety of salts, many of which are highly soluble in water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

Zinc and its salts are highly persistent in water, with half-lives greater than 200 days. The half-life of a pollutant is the amount of time it takes for one-half of the chemical to be degraded.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of zinc found in fish tissues is expected to be considerably higher than the average concentration of zinc in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA.

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Polycyclic Aromatic Hydrocarbons

Steven Pike. M.D.

Polycyclic aromatic hydrocarbons (PAHs) belong to a class of chemicals characterized by molecules containing three or more fused unsaturated carbon rings. PAHs are ubiquitous in the environment and are formed during the process of incomplete combustion or pyrolysis of organic matter. The burning of oil, gas, and other fossil fuels constitutes the main sources of PAHs emitted in the atmosphere. Hundreds of different PAHs are formed in this way as well as heterocyclic or polynuclear aromatic hydrocarbons (PNAs), which are PAHs containing elements other than carbon in their ring structure, such as sulfur, oxygen, or nitrogen. Those compounds containing four to six rings present in PAH mixtures may be carcinogenic.

These compounds have relatively high molecular weights and exist in solid form at room temperature most commonly as condensates on particles or surfaces. They are practically insoluble in water but are soluble in organic solvents. PAHs entering the atmosphere condense when hot combustion gases cool, and form very small particles that can be adsorbed onto existing particles. In addition, PAHs can be formed on surfaces or deep inside anic matter undergoing incomplete pyrolysis. PAH aerosols be transported great distances by winds. PAHs are found in coal, charcoal, automobile exhaust, tobacco smoke, tar, oil, smoked foods, sewage sludge, flue gases, etc. Atmospheric levels vary but are typically higher in urban regions compared with rural, restaurants compared with general office environments, and winter compared with summer. The composition of PAH emissions varies with the source and location, so comparisons are often made by reference to concentrations of benzo(a)pyrene (BaP), which is used as a surrogate or index compound for all the other PAHs contained in the mixture.

Benzo(a)pyrene is not a consistently reliable surrogate for comparing PAH mixtures because the relative concentrations of BaP to other constituents in different mixtures from different sources can vary widely. For example, in cigarette smoke condensate, BaP comprises approximately 1% of the carcinogenic effect, but in extracts of sewage sludge BaP accounts for nearly 23%.

ENVIRONMENTAL SOURCES

Over 97% of the estimated BaP emissions are attributed to stationary fuel combustion—refuse fires, residential furnaces, and coke ovens contributing the largest share, over 87% of the total. A review of PAHs in the environment has been published by Edwards (1983) (1). Tables 112.1 and 112.2 list some common sources of airborne PAH emissions. PAH concentrations in air range from 0.1 ng/m^3 (detection limit) to about 100 ng/m^3 (2, 3). Average urban PAH concentrations in selected cities have been reported as approximately 2.17 ng/m^3 (4). Sawicki (1960) (5) reported BaP air concentrations in urban centers to e from 0.1–61.0 ng/m^3, and in nonurban regions to range 0.01–1.9 ng/m^3. Using BaP as a rough index of exposure, generally heavily polluted air (6) contains approximately 100 ng/m^3, drinking water (7) contains approximately 27 ng/l, and smoked foods (8) contain 100 µg/kg.

Large concentrations of PAHs exist as waste deposits throughout developed countries where manufactured gas plants produced gas for lighting and heating from coal or oil. These plants were in common operation from the mid 1800s until the early 1950s, when they were phased out by the introduction of interstate natural gas pipelines. It is estimated that there were over 1000 such plants throughout the United States prior to World War II, with most concentrated in the Midwest and East (9). The major classes of chemicals associated with gas plant wastes are PAHs, phenolics, volatile organic hydrocarbons (VOCs), various inorganic sulfur and nitrogen species, and, to a lesser extent, trace metals. Table 112.3 lists some published concentrations of BaP detected in soil and water in various geographic locations. Surface water concentrations are reported to range from 0.6–114 ng/l (10).

PAHs are present in foods as a result of biosynthesis, adsorption of particulates on leafy surfaces from atmospheric

Table 112.1. Emission Sources (Tons/y)

Source	BaP Emissions	% of total
Wood burning fireplaces, etc.	25	2.8
Coal refuse fires	310	34.7
Residential furnaces	300	33.6
Coke production	170	19.0
Mobile sources, gasoline	11	1.2
Forest and agricultural refuse burning	11	1.2
Open refuse burning	11	1.2
Vehicle disposal open burning	25	2.8

Source: U.S. Environmental Protection Agency. Preferred Standards Path Report for Polycyclic Organic Matter. Durham, N.C.: U.S. Environ. Prot. Agency, Office Air Qual. Plan. Stand., Strategies Air Stand. Div., 1974. Adapted from adaptation by: Baum EJ. Occurrence and surveillance of polycyclic aromatic hydrocarbons. Polycyclic hydrocarbons and cancer. Vol 1. Orlando, FL: Academic Press 1978.

Table 112.2. Environmental Sources

Exposure Category	BaP (µg/m^3)
Cigarettes unfiltered (1 pack/day)	0.7 µg/day
Cigarettes filtered (1 pack/day)	0.4 µg/day
Airline cockpits (prior to nonsmoking regulations)	
Transatlantic	0.093 (8-hr TWA)
Domestic	0.138 (8-hr TWA)
Coke oven workers	
Topside	18 (8-hr TWA)
Side and bench	7 (8-hr TWA)
Roof tarring	14
Sidewalk tarring	78
Restaurant	0.8 µg/day
	0.03–0.14

Adapted from: Bridbord K, Finlea JF, Wagoner JK, Moran JB, Caplan P. Human exposure to polynuclear aromatic hydrocarbons. Freudenthal RI, Jones PW, ed. Carcinogenesis. Vol 1. Polynuclear aromatic hydrocarbons: chemistry, metabolism, and carcinogenesis. New York, Raven Press, 1976

Table 112.3. Soil and Water BaP Concentrations (μg/kg)

Source	BaP (μg/kg)
Forest	up to 1300*
Nonindustrial sites	up to 127
Towns and vicinities	up to 939
Soils near traffic	up to 2000
Near oil refinery	200.000
Near airport	785
Contaminated by coal tar pitch	685.000
Remote areas	10 to 20

Source: World Health Organization. Monograph on the evaluation of carcinogenic risks of the chemical to man. Certain polycyclic aromatic hydrocarbons and heterocyclic compounds. Vol. 3. Geneva: International Agency Research on Cancer. WHO. 1973. As adapted by: Baum EJ. Occurrence and surveillance of polycyclic aromatic hydrocarbons. Polycyclic hydrocarbons and cancer. Vol. 1. Orlando, FL: Academic Press. 1978.
*Seldom exceeding 10–20 μg/kg in remote areas.

Table 112.4. PAH in Food (μg/kg dried material)

Source	BaP	Chrysene	Benzanthracene
Cereals	0.25–0.84	0.8–14.5	0.4–6.8
Salad	2.8–5.3	5.7–26.5	4.6–15.4
Spinach	7.4	28.0	16.1
Tomatoes	0.22	0.5	0.3
Refined oils/fats	0.9–15	0.5–129	0.5–13.5
Broiled meat/fish	0.2–162	0.5–25.4	0.2–31
Smoked meat/fish	0.2–107	0.3–123	0.02–189
Roasted coffee	0.1–4	0.6–19.1	0.5–14.2
Tea	3.9–21.3	4.6–6.3	

Source: Grimmer G. Carcinogenic hydrocarbons in the human environment. Dtsch. Aptoth. Ztg. 108:529. 1968. Shabad LM. Cohan YL. Contents of benzo(a)pyrene in some crops. Arch. Geschwulstforsch. 40:237. 1972. World Health Organization. Monograph on the evaluation of carcinogenic risks of the chemical to man. Certain polycyclic aromatic hydrocarbons and heterocyclic compounds. Vol 3. Geneva: International Agency for Research in Cancer. WHO. As adapted by: Baum EJ. Occurrence and surveillance of polycyclic aromatic hydrocarbons. Polycyclic hydrocarbons and cancer. Vol 1. Orlando. FL: Academic Press. 1978.

fallout, or more significantly as a result of the processing or cooking of foods prior to consumption. Concentrations are higher on plant surfaces compared with internal tissue, and above ground plants have much higher concentrations than below ground plants. Broad-leafed plants contain more PAHs than thin-leafed, and, while most of this PAH in plants is from atmospheric deposition, washing plants with water is not an effective method for removing PAH contamination from vegetables. Concentrations of BaP in vegetation range from 0.1–150 μg/kg. Wang and Meresz (1981) (11) reported vegetation: soil BaP ratios to range from 0.0001–0.085, while Shabad (1971) (12) and Fritz (1971) (13) reported soil concentrations worldwide to range from 100–1000 μg/kg, with total PAH to typically be 10 times the BaP concentration. Their vegetation:soil ratios were reported to range from 0.002–0.33 for BaP and 0.001–0.183 for total PAH. Some terrestrial plants can take up and translocate PAHs through roots and leaves. The rate of uptake is dependent on physical factors of PAH deposition, soil type and condition, and plant species. Plants can synthesize BaP, and this has been reported to occur during germination of beech, oak, tobacco, wheat, rye, and lentils with concentrations ranging from 10–20 μg/kg of dried material (14). Solid residues from water and sewage treatment plants are also high in PAH content (15–17). Table 112.4 lists some common foods and the reported concentrations of benzo(a)pyrene, chrysene, and benzanthracene in μg/kg of dried material.

METABOLISM

PAHs are metabolized by the cytochrome P450/448-dependent microsomal enzyme system, aryl hydrocarbon hydroxylase (AHS), to form epoxides. The epoxides formed are then converted by epoxide hydrolases to dihydrodiols and diol-epoxides. For BaP metabolism by AHH, the 7,8-epoxide is mainly formed which is considered to be a proximate carcinogen (18). BaP 7,8-epoxide can then be converted to BaP 7,8-dihydrodiol, which can itself be oxidatively metabolized by AHH to the BaP 7,8-diol-9,10-epoxide. Several enantiomeric diol-epoxides are possible, demonstrating wide variations in tumorigenic potency. The wide variability in potency of the enantiomeric forms of diol-epoxides led to Jarina et al. proposing the "bay region" theory to assist in understanding the carcinogenicity of PAHs (19, 20). The "bay region" theory predicted that epoxides located on saturated angular rings in the bay region of a PAH should be highly reactive.

In addition to epoxidation and epoxide conversion to dihydrodiols, a number of other metabolic pathways for PAHs have been observed. Phenols, quinones, sulfates, glucuronates, and reactions with DNA, proteins, and glutathione have been reported (21).

TOXICITY

There are few published reports regarding the acute toxicity of PAHs in animals. Some reports do exist concerning the acute toxicity of specific compounds, but there is little to no information regarding the acute toxicity of PAH mixtures. In general the acute toxicity of PAHs increases as the molecular weight increases and with increasing side chain alkyl substitution on the aromatic nucleus. In animals the acute toxicity of oral or dermal exposure is relatively low, based on experiments on mice and rats. However, repeated exposure to some low dose PAHs resulted in carcinogenicity to these animals. As little as a few micromoles of benzo(a)pyrene were sufficient to cause cancer in less than 6 months in mice repeatedly dosed (22). Chronic exposure to low concentrations of PAHs in water can result in decreased survival, behavioral changes, reproductive effects, and cancer in some sensitive aquatic species (23). The proportionate concentrations of specific compounds in PAH mixtures vary considerably by source, which compounds the problem of evaluating acute toxicity of PAH mixtures. PAHs appear to be mildly to moderately toxic to humans and animals by acute exposure, but in animals chronic exposures to certain PAHs result in cancer. Air pollution studies demonstrating an excess in lung cancer among workers exposed to PAHs from coal gas, tar, coke oven emissions, and soot strongly suggest that PAHs are carcinogenic to humans (24–33). Published reports of acute toxicity studies for the various PAHs are limited.

PAHs produced systemic toxicity manifested by inhibition of growth in mice and rats at doses above 150 mg/kg (34). Rapidly proliferating tissues such as bone marrow, intestinal epithelium, lymphoid tissues, and reproductive organs appear

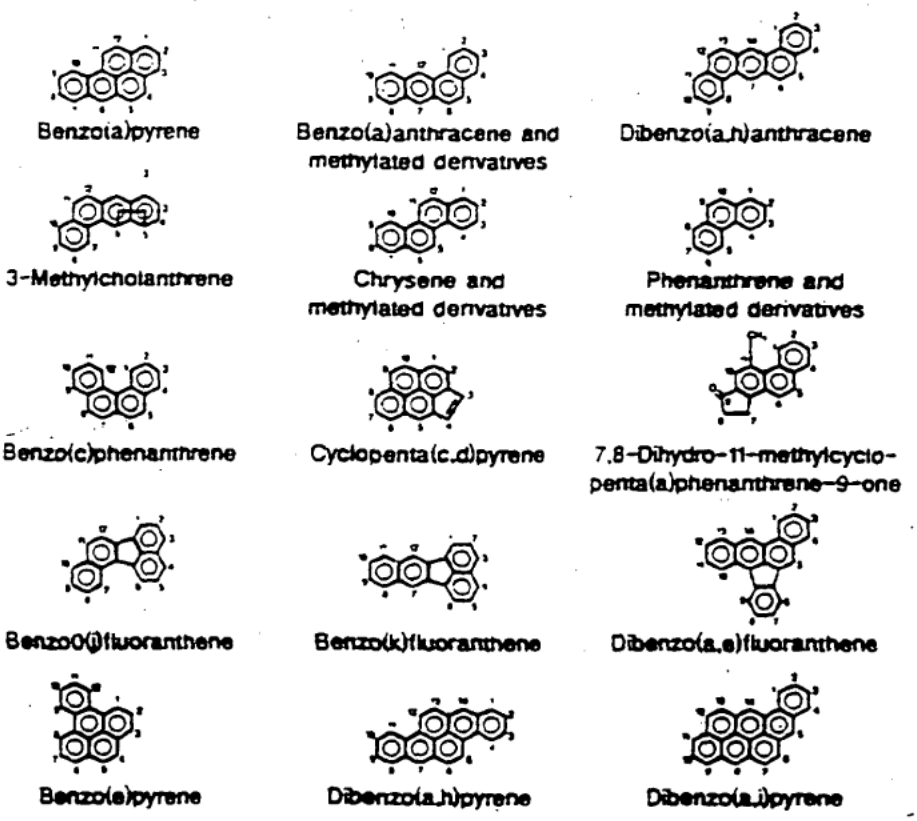

Figure 112.1. Common chemical structures polycyclic aromatic hydrocarbon.

to be most susceptible to PAH toxicity. Specific effects that have been observed in rats fed dimethylbenzanthracene at doses between 50 and 300 mg/kg include: agranulocytosis, anemia, lymphopenia, pancytopenia, and testicular degeneration (35). In mice, thymic degeneration, impaired thyroid development, general wasting, and immunosuppression have been observed at doses of 3-methylcholanthrene (3MC) over 150 mg/kg (36, 37). Some of the PAHs have also been shown to be mutagenic and teratogenic in in vitro test systems and in rodents, respectively. Examination of Table 112.5, which presents the LD_{50} values for several PAH compounds, reveals the lack of acute toxicity data for many of these compounds.

REGULATIONS AND STANDARDS

The 1990–91 American Conference of Governmental Industrial Hygienists' (ACGIH) threshold limit value (TLV) for coal tar pitch volatiles is 0.2 mg/m^3. In the early 1970s the USSR issued a maximum allowable concentration (MAC) for benzo(a)pyrene of 0.15 μg/m^3. The USSR has proposed a 1-ng/m^3 BaP standard for ambient air and a 150-ng/m^3 limit for work environments (38). Sweden issued a TLV of 10 μg/m^3 in 1978 for benzo(a)pyrene and lowered it to 5 μg/m^3 in 1982. Limits for PAH concentrations in drinking water have been recommended by the World Health Organization (WHO) as 7.5 ng/l for BaP and 30 ng/l for total carcinogenic PAHs. The Safe Drinking Water Committee of the National Academy of Sciences was unable to determine a Suggested No Adverse Effect Level (SNARL) for PAHs because of their proven carcinogenicity in animals and their suspected carcinogenicity in humans. Insufficient data were cited for their inability to set 7-day and 24-hour PAH exposure limits for humans (39).

Table 112.5. Acute Toxicity LD_{50} mg/kg[a]

PAH	ORL Rat	ORL Mouse	SQ Rat	SQ Mouse	IVN Rat	IVN Mouse
7,12-Dimethylbenzanthracene	327	340	—	—	54	—
Benzanthracene	—	—	—	—	—	10[b]
Chrysene	—	—	—	—	—	—
Benzo(a)pyrene	—	—	50	—	—	—
Phenanthrene	—	700	—	—	—	56
Anthracene	—	—	—	—	—	—
Pyrene	—	—	—	—	—	—
Naphthalene[c]	1780	—	—	969	—	100
Acenaphthylene	—	—	—	—	—	—
Benzo(j)fluoranthene	—	—	—	—	—	—
Benzo(k)fluoranthene	—	—	—	—	—	—
Benzo(b)fluoranthene	—	—	—	—	—	—
Benzo(ghi)perylene	—	—	—	—	—	—
Benzo(c)phenanthrene	—	—	—	—	—	—

[a]Source: NIOSH Registry of Toxic Effects of Chemical Substances. Taken RL. Lewis Sr. RJ. eds. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health. 1983.
[b]LD_{LO}.
[c]Oral-child LD_{LO}. 100 mg/kg: unk. man LD_{LO} = 74 mg/kg.

REFERENCES

1. Edwards NT. Polycyclic aromatic hydrocarbons (PAH's) in the terrestrial environment—a review. J Environ Qual 1983;12(4):427–441.

2. Sawicki E. Elbert WC. Hauser TR. Fox FT. Stanley TW. Benzo(a)pyrene content of the air of American communities. J Am Ind Hyg Assoc 1960;21:443.

3. Sawicki E. Hauser TR. Elbert WC. Fox FT. Meeker JE. Polynuclear aromatic hydrocarbon composition of the atmosphere in some large American cities. J Am Ind Hyg Assoc 23:137.

4. United States Environmental Protection Agency. Preferred standards path report for polycyclic organic matter. Durham, North Carolina: U.S. Environ. Prot. Agency, Office Air Qual. Plan. Stand., Strategies Air Stand. Div. 1974.

5. Sawicki E. Elbert WC. Hauser TR. Fox FT. Stanley TW. Benzo(a)pyrene content of the air of American communities. J Am Ind Hyg Assoc 1960;21:443.

6. Sawicki E. Hauser TR. Elbert WC. Fox FT. Meeker JE. Polynuclear aromatic hydrocarbon composition of the atmosphere in some large American cities. J Am Ind Hyg Assoc 1962;23:137.

7. Sawicki E. Corey RC. Dooley AE. Giselard JB. Monkman JL. Nelligamma RE. Ripperton LA. Tentative method of spectrophotometric analysis for benzo(a)pyrene in atmospheric particulate matter. Health Lab Sci 7 (suppl 68) (1970).

8. Wierzchowski J. Gajewska R. Determination of 3,4-benzopyrene in smoked fish. Bromatol Chem Toksykol 1972;5:481.

9. Handbook on manufactured gas plant sites. Prepared for: Utility Solid Waste Activities Group, Superfund Committee, Washington, DC by Environmental Research and Technology, Inc. and Koppers Company, Inc., Pittsburgh, PA: Edison Electric Institute, 1984.

10. Sawicki E. Corey RC. Dooley AE. Giselard JB. Monkman JL. Nelligamma RE. Ripperton LA. Tentative method for spectrophometric analysis for benzo(a)pyrene in atmospheric particulate matter. Health Lab Sci 1970; 7 (suppl 68).

11. Wang DT. Meresz O. Occurrence and potential uptake of polynuclear aromatic hydrocarbons of highway traffic origin by proximally grown food crops. (Abstract). Sixth Int. Symp. on PAH. Columbus, OH: Battelle Columbus Laboratory, 1981.

12. Shabad LM. Cohan YL. Ilnitsky AP. Khesina AYA. Shcherbak NP. Smirnov GA. The carcinogenic hydrocarbon benzo(a)pyrene in the soil. J Natl Cancer Inst 1971;47:1179–1191.

13. Fritz W. Extent and sources of contamination of our food with carcinogenic hydrocarbons. Ernaehrungsforschung 1971;16:547–557.

14. Graef W. Diehl H. The natural normal levels of carcinogenic polycyclic aromatic hydrocarbons and the reasons therefore. Arch Hyg Bakteriol 1966;150:49.

15. Borneff J. Fischer R. Carcinogenic substances in water and soil. IX. Investigations on filter mud of a lake water (treatment) plant for PAH. Arch Hyg Bakteriol 1962;146:183.

16. Borneff J. Fischer R. Carcinogenic substances in water and soil. XII. PAH in surface waters. Arch Hyg Bakteriol 1963;146:572.

17. Borneff J. Kunte H. Carcinogenic substances in water and soil. XVII. About the origin and evaluation of PAH in water. Arch Hyg Bakteriol 1965;149:226.

18. Levin W. Wood AW. Yagi H. Dansette PM. Jerina DM. Conney AH. Carcinogenicity of benzo(a)pyrene 4,5-, 7,8-, and 9,10-oxides on mouse skin. Proc Natl Acad Sci USA 1976;73:243.

19. Jerina DM. Daly JW. Oxidation at carbon. In: Parke DV. Smith RL, eds. Drug metabolism. London: Taylor and Francis, Ltd., 1976.

20. Jerina DM. Lehr RE. Yagi H. et al. Mutagenicity of benzo(a)pyrene derivatives and the description of a quantum mechanical model which predicts the ease of carbonium ion formation from diol epoxides. In: DeSerres FJ, F JR. Bend JR. Philpot RM. eds. Vitro metabolic activation and mutage testing. Amsterdam: Elsevier/North-Holland Biomedical, 1976.

21. Pott F. Oberdorster G. Intake and distribution of PAH. In: Grimmer G. ed. Environmental carcinogens: polycyclic aromatic hydrocarbons. Boca Raton, FL: CRC Press, 1983.

22. Rigdon RH. Neal J. Relationship of leukemia to lung and stomach tumors in mice fed benzo(a)pyrene. Proc Soc Exp Biol 1969;130:146.

23. Neff JM. Polycyclic aromatic hydrocarbons in the aquatic environment: sources, fates and biological effects. London: Applied Science Publishers, 1979.

24. Doll R. Fisher REW. Gammon EJ. Gunn W. Hughes GO. Tyrer FH. Wilson W. Mortality of gasworkers with special reference to cancers of the lung and bladder, chronic bronchitis, and pneumoconiosis. Br J Ind Med 1965;22:1–12.

25. Doll R. Vessey MP. Beasley RWR. et al. Mortality of gasworkers—final report of a prospective study. Br J Ind Med 1972;29:394–406.

26. Hammond EC. Selikoff IJ. Lawther PL. Seidman H. Inhalation of benzpyrene and cancer in man. Ann NY Acad Sci 1976;271:116–124.

27. Henry SA. Kennaway NM. Kennaway EL. The incidence of cancer of the bladder and prostate in certain occupations. J Hyg 1931;31:125–137.

28. Kawai M. Amamoto H. Harada K. Epidemiologic study of occupational lung cancer. Arch Environ Health 1967;14:859–864.

29. Kuroda S. Occupational pulmonary cancer of generator gas workers. Ind Med 1937;6:304–306.

30. Mazumdar S. Redmond C. Sollecito W. and Sussman N. An epidemiological study of exposure to coal tar pitch volatiles among coke oven workers. J Air Pollut Cont Assoc 1975;25:382–389.

31. Redmond CK. Ciocco A. Lloyd JW. Rush HW. Long term mortality study of steelworkers. VI. Mortality from malignant neoplasms among coke oven workers. J Occup Med 1972;14:621–629.

32. Redmond CK. Strobino BR. Cypress RH. Cancer experience among coke by-product workers. Ann NY Acad Sci 1976;271:102–115.

33. Reid DD. Buck C. Cancer in coking plant workers. Br J Ind Med 1956;13:265–269.

34. Haddow A. Scott CM. Scott JD. The influence of certain carcinogenic and other hydrocarbons on body growth in the rat. Proc R Soc Lond Ser 1937;122:477–507.

35. Philips FS. Sternberg SS. Marquardt H. In vivo cytotoxicity of polycyc. hydrocarbons. In: Loomis TA. ed. Pharmacology and the future of man. toxicological problems. Proceedings of the Fifth International Congress on pharmacology. San Francisco, CA. July 23–28. 1972. 1973;2:75–88.

36. Malmgren RA. Bennison BE. McKinley Jr. TW. Reduced antibody titers in mice treated with carcinogenic and cancer chemotherapeutic agents. Proc Soc Exp Biol Med 1952;79:484–488.

37. Yasuhira K. Damage to the thymus and other lymphoid tissues from 3-methylcholanthrene, and subsequent thymoma production, in mice. Cancer Res 1964;24:558–569.

38. Shabad LM. On the so-called MAC (maximal allowable concentrations) for carcinogenic hydrocarbons. Neoplasma 1972;22:459.

39. National Academy of Sciences. Drinking Water and Health. Vol 4. Safe Drinking Water Committee. Board on Toxicology and Environmental Health Hazards. Assembly of Life Sciences. National Research Council. Washington, DC: National Academy Press, 1982.

Common Name: Polychlorinated Biphenyls
CAS Number: 1336-36-3
DOT Number: UN 2315
Date: May, 1989

HAZARD SUMMARY

* Polychlorinated Biphenyls can affect you when breathed in and by passing through your skin.
* Polychlorinated Biphenyls are CARCINOGENS HANDLE WITH EXTREME CAUTION.
* They may be teratogens and may damage the adult reproductive system.
* Exposure can cause an acne like skin rash (called chloracne).
* They can damage the liver.
* High exposure can damage the nervous system, causing numbness, weakness and tingling ("pins and needles") in the arms and legs.

IDENTIFICATION

Polychlorinated Biphenyls are a mixture of chemicals that are clear to yellow oily liquids or solids. They are used in insulating fluids of electrical systems.

REASON FOR CITATION

* Polychlorinated Biphenyls are on the Hazardous Substance List because they are regulated by OSHA and cited by NIOSH, DOT, IARC, NTP, DEP and EPA.
* These chemicals are on the Special Health Hazard Substance List because they are CARCINOGENS and TERATOGENS.
* Definitions are attached.

HOW TO DETERMINE IF YOU ARE BEING EXPOSED

* Exposure to hazardous substances should be routinely evaluated. This may include collecting personal and area air samples. You can obtain copies of sampling results from your employer. You have a legal right to this information under OSHA 1910.20.
* If you think you are experiencing any work related health problems, see a doctor trained to recognize occupational diseases. Take this Fact Sheet with you.

WORKPLACE EXPOSURE LIMITS

OSHA: The legal airborne permissible exposure limit (PEL) is 1 mg/m3 (42% Chlorine) and 0.5 mg/m3 (54% Chlorine) averaged over an 8 hour workshift.

NIOSH: The recommended airborne exposure limit is 0.001 mg/m3 averaged over a 10 hour workshift.

* The above exposure limits are for air levels only. When skin contact also occurs, you may be overexposed, even though air levels are less than the limits listed above.
* Polychlorinated Biphenyls are PROBABLE CANCER CAUSING AGENTS in humans. There may be no safe level of exposure to carcinogens, so all contact should be reduced to the lowest possible level.

WAYS OF REDUCING EXPOSURE

* Where possible, enclose operations and use local exhaust

ventilation at the site of chemical release. If local exhaust ventilation or enclosure is not used, respirators should be worn.
* A regulated, marked area should be established where Polychlorinated Biphenyls are handled, used, or stored as recommended by NIOSH.
* Wear full body protective work clothing.
* Wash thoroughly immediately after exposure to Polychlorinated Biphenyls and on exit from the work area.
* Post hazard and warning information in the work area. In addition, as part of an ongoing education and training effort, communicate all information on the health and safety hazards of Polychlorinated Biphenyls to potentially exposed workers.

This Fact Sheet is a summary source of information of all potential and most severe health hazards that may result from exposure. Duration of exposure, concentration of the substance and other factors will affect your susceptibility to any of the potential effects described below.

HEALTH HAZARD INFORMATION

Acute Health Effects
The following acute (short term) health effects may occur immediately or shortly after exposure to Polychlorinated Biphenyls:

* Exposure to the vapor can irritate the eyes, nose and throat.
* High exposures can damage the liver.

Chronic Health Effects
The following chronic (long term) health effects can occur at some time after exposure to Polychlorinated Biphenyls and can last for months or years:

Cancer Hazard
* Polychlorinated Biphenyls are PROBABLE CARCINOGENS in humans. There is some limited evidence that they cause skin cancer in humans and they have been shown to cause liver cancer in animals.
* Many scientists believe there is no safe level of exposure to a CARCINOGEN. Such substances may also have the potential for causing reproductive damage in humans.

Reproductive Hazard
* Polychlorinated Biphenyls may be TERATOGENS in humans since they have been shown to be teratogens in animals.
* They may be passed to a child through mother's milk.
* Polychlorinated Biphenyls can affect the reproductive system of adults.

Other Long Term Effects
* Repeated exposures can cause liver damage.
* Polychlorinated Biphenyls can cause a severe acne like rash (chloracne). This may persist for years.
* High exposures can damage the nervous system, causing numbness, weakness, and tingling ("pins and needles") in the arms and legs.

MEDICAL

Medical Testing
Before beginning employment and at regular times after that, the following are recommended:

* Liver function tests.
* Serum triglycerides level.
* Exam of the skin.

If symptoms develop or overexposure is suspected, the following may be useful:

* Blood PCB levels.
* Nerve conduction studies should be considered.

Any evaluation should include a careful history of past and present symptoms with an exam. Medical tests that look for damage already done are not a substitute for controlling exposure.

Request copies of your medical testing. You have a legal right to this information under OSHA 1910.20.

Mixed Exposures
Because more than light alcohol consumption can cause liver damage, drinking alcohol can increase the liver damage caused by Polychlorinated Biphenyls.

WORKPLACE CONTROLS AND PRACTICES

Unless a less toxic chemical can be substituted for a hazardous substance, ENGINEERING CONTROLS are the most effective way of reducing exposure. The best protection is to enclose operations and/or provide local exhaust ventilation at the site of chemical release. Isolating operations can also reduce exposure. Using respirators or protective equipment is less effective than the controls mentioned above, but is sometimes necessary.

in place for highly toxic chemicals or when significant skin, eye, or breathing exposures are possible.

In addition, the following controls are recommended:

* Where possible, automatically transfer Polychlorinated Biphenyls from drums or other storage containers to process containers.
* Specific engineering controls are recommended for this chemical by NIOSH. Refer to the NIOSH criteria document: Occupational Exposure to Polychlorinated Biphenyls #77 225.

Good WORK PRACTICES can help to reduce hazardous exposures. The following work practices are recommended:

* Workers whose clothing has been contaminated by Polychlorinated Biphenyls should change into clean clothing promptly.
* Do not take contaminated work clothes home. Family members could be exposed.
* Contaminated work clothes should be laundered by individuals who have been informed of the hazards of exposure to

Polychlorinated Biphenyls.

* If there is the possibility of skin exposure, emergency shower facilities should be provided.
* On skin contact with Polychlorinated Biphenyls, immediately wash or shower to remove the chemical. At the end of the workshift, wash any areas of the body that may have contacted Polychlorinated Biphenyls, whether or not known skin contact has occurred.
* Do not eat, smoke, or drink where Polychlorinated Biphenyls are handled, processed, or stored, since the chemicals can be swallowed. Wash hands carefully before eating or smoking.
* If solid, when vacuuming, a high efficiency particulate absolute (HEPA) filter should be used, not a standard shop vacuum.

PERSONAL PROTECTIVE EQUIPMENT

WORKPLACE CONTROLS ARE BETTER THAN PERSONAL PROTECTIVE EQUIPMENT. However, for some jobs (such as outside work, confined space entry, jobs done only once in a while, or jobs done while workplace controls are being installed), personal protective equipment may be appropriate.

The following recommendations are only guidelines and may not apply to every situation.

Clothing

* Avoid skin contact with Polychlorinated Biphenyls. Wear protective gloves and clothing. Safety equipment suppliers/ manufacturers can provide recommendations on the most protective glove/ clothing material for your operation.
* All protective clothing (suits, gloves, footwear, headgear) should be clean, available each day, and put on before work.
* Viton is recommended as a good protective material.

Eye Protection

* Eye protection is included in the recommended respiratory protection.

Respiratory Protection

IMPROPER USE OF RESPIRATORS IS DANGEROUS. Such equipment should only be used if the employer has a written program that takes into account workplace conditions, requirements for worker training, respirator fit testing and medical exams, as described in OSHA 1910.134.

* At any exposure level, use a MSHA/NIOSH approved supplied air respirator with a full facepiece operated in the positive pressure mode or with a full facepiece, hood, or helmet in the continuous flow mode, or use a MSHA/NIOSH approved self contained breathing apparatus with a full facepiece operated in pressure demand or other positive pressure mode.

Common Name: Polychlorinated Biphenyls
DOT Number: UN 2315
DOT Emergency Guide code: 15
CAS Number: 1336-36-3

Hazard rating NJ DOH NFPA

FLAMMABILITY	Not Found	Not Rated
REACTIVITY	Not Found	Not Rated

CARCINOGEN
POISONOUS GASES ARE PRODUCED IN FIRE

Hazard Rating Key: 0=minimal; 1=slight; 2=moderate; 3=serious; 4=severe

FIRE HAZARDS

* Polychlorinated Biphenyls may burn, but do not readily ignite.
* Use dry chemical, CO2, water spray, or foam extinguishers.
* POISONOUS GASES ARE PRODUCED IN FIRE, including Dioxin and Chlorinated Dibenzofurans.
* If employees are expected to fight fires, they must be trained and equipped as stated in OSHA 1910.156.

SPILLS AND EMERGENCIES

If Polychlorinated Biphenyls are spilled or leaked, take the following steps:

* Restrict persons not wearing protective equipment from area of spill or leak until clean up is complete.
* Ventilate the area of spill or leak.
* Absorb liquids in vermiculite, dry sand, earth, or a similar material and deposit in sealed containers.
* Collect powdered material in the most convenient and safe manner and deposit in sealed containers.
* It may be necessary to contain and dispose of Polychlorinated Biphenyls as a HAZARDOUS WASTE. Contact your State Environmental Program for specific recommendations.

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FOR LARGE SPILLS AND FIRES immediately call your fire department.

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HANDLING AND STORAGE

* Prior to working with Polychlorinated Biphenyls you should be trained on their proper handling and storage.
* Store in tightly closed containers in a cool well ventilated area away from STRONG OXIDIZERS (such as CHLORINE, BROMINE, and FLUORINE).
* Polychlorinated Biphenyls should be handled only in an established, controlled, regulated area.

FIRST AID
POISON INFORMATION

Eye Contact
* Immediately flush with large amounts of water for at least 15 minutes, occasionally lifting upper and lower lids.

Skin Contact
* Quickly remove contaminated clothing. Immediately wash contaminated skin with large amounts of soap and water.

Breathing

* Remove the person from exposure.
* Begin rescue breathing if breathing has stopped and CPR if heart action has stopped.
* Transfer promptly to a medical facility.

PHYSICAL DATA

Flash Point: 383oF (195oC)
Water Solubility: Slightly soluble

Other Names and Formulations:

This Fact Sheet can be used for the following substances:

PCB 1242 (Chlorodiphenyl 42% Chlorine) CAS # 53469 21 9;
PCB 1254 (Chlorodiphenyl 54% Chlorine) CAS # 11097 69 1.

NEW JERSEY DEPARTMENT OF HEALTH
Right to Know Program
CN 368, Trenton, NJ 08625 0368

ECOLOGICAL INFORMATION

Polychlorinated biphenyls are complex mixtures of chlorobiphenyls which have been marketed for uses according to the percentage of chlorine in the mixture. The lesser chlorinated PCBs are colorless mobile liquids. Increased chlorination produces more viscous liquids, with further chlorination producing sticky resins or white powders. Because of their heat stability, PCBs were commonly used in electrical capacitors and transformers, and industrial heat transfer applications. PCBs may enter the environment from leakage from industrial and electrical equipment, from industrial discharges, spills, leaching from municipal landfills, and from previously contaminated sediments.

ACUTE (SHORT-TERM) ECOLOGICAL EFFECTS

Acute toxic effects may include the death of animals, birds, or fish, and death or low growth rate in plants. Acute effects are seen two to four days after animals or plants come in contact with a toxic chemical substance.

Polychlorinated biphenyls have high acute toxicity to aquatic life. Insufficient data are available to evaluate or predict the short-term effects of PCBs to plants, birds, or land animals.

CHRONIC (LONG-TERM) ECOLOGICAL EFFECTS

Chronic toxic effects may include shortened lifespan, reproductive problems, lower fertility, and changes in appearance or behavior. Chronic effects can be seen long after first exposure(s) to a toxic chemical.

Polychlorinated biphenyls have high chronic toxicity to aquatic life. Insufficient data are available to evaluate or predict the long-term effects of PCBs to plants, birds, or land animals.

WATER SOLUBILITY

Polychlorinated biphenyls are slightly soluble in water. Concentrations of less than 1 milligram will mix with a liter of water.

DISTRIBUTION AND PERSISTENCE IN THE ENVIRONMENT

The relative distribution of the various PCBs depends on the level of chlorination. Some PCBs will probably be highly persistent in water, with half-lives greater than 200 days. Potential PCB distribution in the various environmental compartments can have the following ranges, depending on degree of chlorination: air, 0-34%; terrestrial soils, 33-52%; water, 0-1.8%; suspended solids, 0.05-0.08%; aquatic biota, 0.02-0.03%; aquatic sediments, 30-50%.

BIOACCUMULATION IN AQUATIC ORGANISMS

Some substances increase in concentration, or bioaccumulate, in living organisms as they breathe contaminated air, drink contaminated water, or eat contaminated food. These chemicals can become concentrated in the tissues and internal organs of animals and humans.

The concentration of polychlorinated biphenyls found in fish tissues is expected to be considerably higher than the average concentration of PCBs in the water from which the fish was taken.

SUPPORT DOCUMENT: AQUIRE Database, ERL-Duluth, U.S. EPA., EEB OCB risk document,

*** CHEMICAL IDENTIFICATION ***

RTECS NUMBER : WH8675000
CHEMICAL NAME : Stannane, tri-n-butyl-, hydride
CAS REGISTRY NUMBER : 688-73-3
BEILSTEIN REFERENCE NO. : 3587329
REFERENCE : 4-04-00-04312 (Beilstein Handbook Reference)
LAST UPDATED : 199712
DATA ITEMS CITED : 25
MOLECULAR FORMULA : C12-H28-Sn
MOLECULAR WEIGHT : 291.09
WISWESSER LINE NOTATION : 4-SN-H4&4
COMPOUND DESCRIPTOR : Organometallic
SYNONYMS/TRADE NAMES :

* Stannane, tributyl-
* Tin, tri-n-butyl-, hydride
* Tributylstannane
* Tributylstannic hydride
* Tributyltin
* Tributyltin hydride
* Tri-n-butyltin hydride

*** HEALTH HAZARD DATA ***

** ACUTE TOXICITY DATA **

TYPE OF TEST : LCLo - Lowest published lethal concentration
ROUTE OF EXPOSURE : Inhalation
SPECIES OBSERVED : Rodent - mouse
DOSE/DURATION : 1460 mg/m3/10M
TOXIC EFFECTS :
Sense Organs and Special Senses (Eye) - lacrimation
Behavioral - convulsions or effect on seizure threshold
Lungs, Thorax, or Respiration - dyspnea
REFERENCE :
NDRC** National Defense Research Committee, Office of Scientific Research and Development, Progress Report. Volume(issue)/page/year: NDCrc-132,FEB1942

*** REVIEWS ***

ACGIH TLV-TWA 0.1 mg(Sn)/m3;STEL 0.2 mg/m3 (skin)
DTLVS* The Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs) booklet issues by American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, OH, 1996 Volume(issue)/page/year: TLV/BEI,1997

ACGIH TLV-Not classifiable as a human carcinogen
DTLVS* The Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs) booklet issues by American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, OH, 1996 Volume(issue)/page/year: TLV/BEI,1997

*** U.S. STANDARDS AND REGULATIONS ***

MSHA STANDARD-air:TWA 0.1 mg(Sn)/m3
DTLVS* The Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs) booklet issues by American Conference of Governmental Industrial

Hygienists (ACGIH), Cincinnati, OH, 1996 Volume(issue)/page/year: 3,258,1971

OSHA PEL (Gen Indu):8H TWA 0.1 mg(Sn)/m3
CFRGBR Code of Federal Regulations. (U.S. Government Printing Office, Supt. of Documents, Washington, DC 20402) Volume(issue)/page/year: 29,1910.1000,1994

OSHA PEL (Construc):8H TWA 0.1 mg(Sn)/m3
CFRGBR Code of Federal Regulations. (U.S. Government Printing Office, Supt. of Documents, Washington, DC 20402) Volume(issue)/page/year: 29,1926.55,1994

OSHA PEL (Shipyard):8H TWA 0.1 mg(Sn)/m3
CFRGBR Code of Federal Regulations. (U.S. Government Printing Office, Supt. of Documents, Washington, DC 20402) Volume(issue)/page/year: 29,1915.1000,1993

OSHA PEL (Fed Cont):8H TWA 0.1 mg(Sn)/m3
CFRGBR Code of Federal Regulations. (U.S. Government Printing Office, Supt. of Documents, Washington, DC 20402) Volume(issue)/page/year: 41,50-204.50,1994

*** OCCUPATIONAL EXPOSURE LIMITS ***

OEL-AUSTRALIA:TWA 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-BELGIUM:TWA 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-DENMARK:TWA 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-FINLAND:TWA 0.1 mg(Sn)/m3;STEL 0.3 mg(Sn)/m3;Skin JAN 1993

OEL-FRANCE:TWA 0.1 mg(Sn)/m3;STEL 0.2 mg(Sn)/m3 JAN 1993

OEL-GERMANY:TWA 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-HUNGARY:STEL 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-THE NETHERLANDS:TWA 0.1 mg(Sn)/m3;Skin JAN 1993

OEL-THE PHILIPPINES:TWA 0.1 mg(Sn)/m3 JAN 1993

OEL-SWEDEN:TWA 0.1 mg(Sn)/m3;STEL 0.2 mg(Sn)/m3;Skin JAN 1993

OEL-SWITZERLAND:TWA 0.1 mg(Sn)/m3;STEL 0.2 mg(Sn)/m3;Skin JAN 1993

OEL-THAILAND:TWA 0.1 mg(Sn)/m3 JAN 1993

OEL-UNITED KINGDOM:TWA 0.1 mg(Sn)/m3;STEL 0.2 mg(Sn)/m3;Skin JAN 1993

OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA check ACGIH TLV

OEL IN NEW ZEALAND, SINGAPORE, VIETNAM check ACGIH TLV

*** NIOSH STANDARDS DEVELOPMENT AND SURVEILLANCE DATA ***

NIOSH RECOMMENDED EXPOSURE LEVEL (REL) :
NIOSH REL TO ORGANOTIN COMPOUND-air:10H TWA 0.1 mg(Sn)/m3
REFERENCE :

NIOSH* National Institute for Occupational Safety and Health, U.S. Dept. of Health, Education, and Welfare, Reports and Memoranda.
Volume(issue)/page/year: DHHS #92-100,1992

*** STATUS IN U.S. ***

EPA TSCA Section 8(b) CHEMICAL INVENTORY

*** END OF RECORD ***

Z1140 T03 ZINC ACETATE, DIHYDRATE PAGE: 1
EFFECTIVE: 01/04/94 ISSUED: 01/16/94

J.T.BAKER INC., 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865

SECTION I - PRODUCT IDENTIFICATION

PRODUCT NAME: **ZINC ACETATE, DIHYDRATE**
COMMON SYNONYMS: ACETIC ACID, ZINC SALT, DIHYDRATE; ZINC DIACETATE, DIHYDRATE
CHEMICAL FAMILY: ZINC COMPOUNDS
FORMULA: (CH3COO)2ZN 2H2O
FORMULA WT.: 219.51
CAS NO.: **5970-45-6**
NIOSH/RTECS NO.: ZG8750000
PRODUCT USE: LABORATORY REAGENT
PRODUCT CODES: 5023,4304,4298,5658,4296

ZINC ACETATE 5970-45-6

PRECAUTIONARY LABELING

BAKER SAF-T-DATA* SYSTEM

HEALTH	-	1	SLIGHT
FLAMMABILITY	-	1	SLIGHT
REACTIVITY	-	0	NONE
CONTACT	-	2	**MODERATE**

LABORATORY PROTECTIVE EQUIPMENT

GOGGLES; LAB COAT

U.S. PRECAUTIONARY LABELING

CAUTION

CAUSES IRRITATION. MAY BE HARMFUL IF SWALLOWED.
AVOID CONTACT WITH EYES, SKIN, CLOTHING. DO NOT BREATHE DUST. KEEP IN TIGHTLY CLOSED CONTAINER. USE WITH ADEQUATE VENTILATION. WASH THOROUGHLY AFTER HANDLING.

INTERNATIONAL LABELING

DO NOT BREATHE DUST. AVOID CONTACT WITH SKIN AND EYES.

SAF-T-DATA* STORAGE COLOR CODE: ORANGE (GENERAL STORAGE)

CONTINUED ON PAGE: 2

SECTION VI - REACTIVITY DATA

STABILITY: STABLE HAZARDOUS POLYMERIZATION: WILL NOT OCCUR

CONDITIONS TO AVOID: HEAT

INCOMPATIBLES: ALKALIES, STRONG OXIDIZING AGENTS

DECOMPOSITION PRODUCTS: OXIDES OF ZINC, CARBON MONOXIDE, CARBON DIOXIDE

SECTION VII - SPILL & DISPOSAL PROCEDURES

STEPS TO BE TAKEN IN THE EVENT OF A SPILL OR DISCHARGE
WEAR SELF-CONTAINED BREATHING APPARATUS AND FULL PROTECTIVE CLOTHING. WITH CLEAN SHOVEL, CAREFULLY PLACE MATERIAL INTO CLEAN, DRY CONTAINER AND COVER; REMOVE FROM AREA. FLUSH SPILL AREA WITH WATER.

POSAL PROCEDURE
DISPOSE IN ACCORDANCE WITH ALL APPLICABLE FEDERAL, STATE, AND LOCAL ENVIRONMENTAL REGULATIONS.

SECTION VIII - INDUSTRIAL PROTECTIVE EQUIPMENT

VENTILATION: USE ADEQUATE GENERAL OR LOCAL EXHAUST VENTILATION TO KEEP FUME OR DUST LEVELS AS LOW AS POSSIBLE.

RESPIRATORY PROTECTION: NONE REQUIRED WHERE ADEQUATE VENTILATION CONDITIONS EXIST. IF AIRBORNE CONCENTRATION IS HIGH, USE AN APPROPRIATE RESPIRATOR OR DUST MASK.

EYE/SKIN PROTECTION: SAFETY GOGGLES, UNIFORM, RUBBER GLOVES ARERECOMMENDED.

SECTION IX - STORAGE AND HANDLING PRECAUTIONS

SAF-T-DATA* STORAGE COLOR CODE: ORANGE (GENERAL STORAGE)

STORAGE REQUIREMENTS
KEEP CONTAINER TIGHTLY CLOSED. SUITABLE FOR ANY GENERAL CHEMICAL STORAGE AREA.

CONTINUED ON PAGE: 6

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FOR A PARTICULAR PURPOSE.
THE USER SHOULD RECOGNIZE THAT THIS PRODUCT CAN CAUSE SEVERE INJURY AND EVEN DEATH, ESPECIALLY IF IMPROPERLY HANDLED OR THE KNOWN DANGERS OF USE ARE NOT HEEDED. READ ALL PRECAUTIONARY INFORMATION. AS NEW DOCUMENTED GENERAL SAFETY INFORMATION BECOMES AVAILABLE, BAKER WILL PERIODICALLY REVISE THIS MATERIAL SAFETY DATA SHEET.
NOTE: CHEMTREC, CANUTEC, AND NATIONAL RESPONSE CENTER EMERGENCY TELEPHONE NUMBERS ARE TO BE USED ONLY IN THE EVENT OF CHEMICAL EMERGENCIES INVOLVING A SPILL, LEAK, FIRE, EXPOSURE, OR ACCIDENT INVOLVING CHEMICALS. ALL NON-EMERGENCY QUESTIONS SHOULD BE DIRECTED TO CUSTOMER SERVICE (1-800-JTBAKER) FOR ASSISTANCE.

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APPROVED BY QUALITY ASSURANCE DEPARTMENT.

-- LAST PAGE --
"ISSUED BY VWR 04/23/94"

SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

DOMESTIC (D.O.T.)

PROPER SHIPPING NAME: HAZARDOUS SUBSTANCE, SOLID, N.O.S. (ZINC ACETATE, DIHYDRATE)
HAZARD CLASS: ORM-E
UN/NA: NA9188 REPORTABLE QUANTITY: 1000 LBS.
LABELS: NONE
REGULATORY REFERENCES: 49CFR 172.101; 173.500; 173.510

INTERNATIONAL (I.M.O.)

PROPER SHIPPING NAME: CHEMICALS, N.O.S. (NON-REGULATED)
MARINE POLLUTANTS: NO

AIR (I.C.A.O.)

PROPER SHIPPING NAME: CHEMICALS, N.O.S. (NON-REGULATED)

U.S. CUSTOMS HARMONIZATION NUMBER: 29152900007

NOTE: WHEN HANDLING LIQUID PRODUCTS, SECONDARY PROTECTIVE CONTAINERS MUST BE USED FOR CARRYING.
-N/A = NOT APPLICABLE, OR NOT AVAILABLE;
N/E = NOT ESTABLISHED.-
THE INFORMATION IN THIS MATERIAL SAFETY DATA SHEET MEETS THE REQUIREMENTS OF THE UNITED STATES OCCUPATIONAL SAFETY AND HEALTH ACT AND REGULATIONS PROMULGATED THEREUNDER (29 CFR 1910.1200 ET. SEQ.) AND THE CANADIAN WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEM. THIS DOCUMENT IS INTENDED ONLY AS A GUIDE TO THE APPROPRIATE PRECAUTIONARY HANDLING OF THE MATERIAL BY A PERSON TRAINED IN, OR SUPERVISED BY A PERSON TRAINED IN, CHEMICAL HANDLING. THE USER IS RESPONSIBLE FOR DETERMINING THE PRECAUTIONS AND DANGERS OF THIS CHEMICAL FOR HIS OR HER PARTICULAR APPLICATION. DEPENDING ON USAGE, PROTECTIVE CLOTHING INCLUDING EYE AND FACE GUARDS AND RESPIRATORS MUST BE USED TO AVOID CONTACT WITH MATERIAL OR BREATHING CHEMICAL VAPORS/FUMES.
EXPOSURE TO THIS PRODUCT MAY HAVE SERIOUS ADVERSE HEALTH EFFECTS. THIS CHEMICAL MAY INTERACT WITH OTHER SUBSTANCES. SINCE THE POTENTIAL USES ARE SO VARIED, BAKER CANNOT WARN OF ALL OF THE POTENTIAL DANGERS OF USE OR INTERACTION WITH OTHER CHEMICALS OR MATERIALS. BAKER WARRANTS THAT THE CHEMICAL MEETS THE SPECIFICATIONS SET FORTH ON THE LABEL.
BUYER DISCLAIMS ANY OTHER WARRANTIES, EXPRESSED OR IMPLIED WITH REGARD TO THE PRODUCT SUPPLIED HEREUNDER, ITS MERCHANTABILITY OR ITS FITNESS

CONTINUED ON PAGE: 7

ATTACHMENT E-3

TRAINING CERTIFICATION FORMS

Prezant

330 6th Ave. N, Suite 200
Seattle, WA 98109 • (206) 281-8858

Complies with 29 CFR 1910.120 / HAZWOPER Refresher

This card certifies that

Dave Browning

has completed

8 hours of refresher training in
Hazardous Waste Operations

Aug 16, 1999
Certif: (b) (6)
Annual refresher training
required by: ***August 15, 2000***

Lynn Pedone
Training Coordinator

Prezant

330 6th Ave. N, Suite 200
Seattle, WA 98109 • (206) 281-8858

Complies with 29 CFR 1910.120 / HAZWOPER Refresher

This card certifies that

Chris Kirk

has completed

8 hours of refresher training in
Hazardous Waste Operations

Aug 16, 1999
Certif: (b) (6)
Annual refresher training
required by: ***August 15, 2000***

Lynn Pedone
Training Coordinator

Certificate of Completion

This is to certify that

Sandy Browning

has satisfactorily completed

8 hours of refresher training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Feb 26, 1999

Lynn Padovan
Training Administrator

Date Expires Feb 26, 2000

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Vicki Fagerness

has satisfactorily completed

8 hours of refresher training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Mar 26, 1999

Training Administrator

Date Expires Mar 25, 2000

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Gene Revelas

has satisfactorily completed

8 hours of refresher training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Feb 26, 1999

Training Administrator

Date Expires Feb 26, 2000

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Tom Schulz

has satisfactorily completed

8 hours of refresher training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Jun 25, 1999

Lynn Pedone
Training Administrator

Cert. # [redacted]
Conducted at:
Prezant Associates

Date Expires Jun 24, 2000

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Pete Striplin

has satisfactorily completed

8 hours of refresher training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Feb 26, 1999

Training Administrator

Date Expires Feb 26, 2000

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Tom Schultz

has satisfactorily completed
8 hours of supervisor training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Sep 23, 1999

Training Coordinator

Prezant

Cert. # (b) (6)

Conducted for:
Prezant Associates

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Pete Striplin

has satisfactorily completed

8 hours of supervisor training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Apr 29, 1999

Lynn Pedone

Training Administrator

THE NORTHWEST LEADING TRAINING ORGANIZATION

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858

Certificate of Completion

This is to certify that

Dave Browning

has satisfactorily completed

8 hours of supervisor training in

Hazardous Waste Operations

in compliance with OSHA 29 CFR 1910.120

Apr 29, 1999

Lynn Pedone

Training Administrator

Prezant

THE NORTHWEST LEADING TRAINING ORGANIZATION

Cert. # (b) (6)

Conducted at:
Prezant Associates

Prezant Associates, Inc. • 330 Sixth Avenue North, Suite 200 • Seattle, Washington 98109 • (206) 281-8858